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TESTING OF NEW BRUNSWICK OIL SHALE IN R.P.C. TEST FACILITY

by

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Research and Productivity Council

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TESTING OF NEW BRUNSWICK OIL SHALE IN R.P.C. TEST FACILITY

Final Report

Submitted to

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SUMMARY

Retorting trials were carried out on two oil shale samples from the Albert Formation of southeastern New Brunswick. One sample is from the Albert Mines zone (32.4 Imp. Gal/short ton) and, the other is from the Dolomite Marlstone Zone (10.9 Imp. Gal/short ton). High oil recoveries were achieved. For the Albert Mines sample the yield was 101.2% of the Fischer assay while that for the Dolomite Marlstone was even higher at 134.5%.

The product oils are light with an API gravity in the range of 22.7 to 23.9. The oils have relatively low concentrations of nitrogen and sulphur, therefore, would not require extensive hydrotreating (upgrading) prior to their use in a conventional refinery. The distillation characteristics of the product oils indicate that New Brunswick shale oil would probably be a good feedstock for the production of gasoline, kerosen, jet and diesel fuels.

SOMMAIRE

Des tests de pyrogénation ont été effectués sur deux échantillons de schistes bitumineux provenant de la formation Albert située au sud-est du Nouveau-Brunswick. Un des échantillons provient de la zone d'Albert Mines (32,4 gallons impériaux/tonne courte) et l'autre de la zone Dolomite Marlstone (10,9 gallons impériaux/tonne courte). Des taux de récupération élevés de pétrole ont été obtenus. Le rendement de l'échantillon d'Albert Mines, selon le procédé Fischer, a été de 11,2 %, et celui de l'échantillon en provenance de Dolomite Marlstone a été encore plus élevé, soit 134,5 %.

Les hydrocarbures obtenus sont légers avec une densité API variant entre 22,7 et 23,9. Ces produits présentent des concentrations relativement faibles d'azote et de soufre et ne devraient donc pas nécessiter d'hydrotraitement intensif (amélioration) avant d'être envoyés à une raffinerie classique. Les caractéristiques de distillation des produits indiquent que les schistes bitumineux du Nouveau-Brunswick constitueraient probablement une bonne matière première pour la fabrication d'essence, de kérosène, et de carburant pour moteurs à réaction et diesel.

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1.0 INTRODUCTION

The work described in this report was in fulfilment of a contract from the Energy Branch of the Department of Natural Resources and Energy of New Brunswick. The request was to evaluate the physical characteristics and shale oil recoveries for two shale samples. As outlined in the request for proposal which is included in Appendix A, the Department of Natural Resources and Energy, N.B. Power, Canadian Occidental Petroleum Ltd. and EMR are interested in the extraction of hydrocarbons from N.B. oil shale. This project which is funded under the terms of Canada/New Brunswick Mineral Development Agreement (MDA), will provide the Department of Natural Resources and Energy of New Brunswick with some of the necessary data required to assess the possibility of a future oil shale and refining industry in New Brunswick.

The oil shale deposits of the Albert Formation in the Moncton Subbasin, New Brunswick, are considered to be Canada's largest and highest grade oil shale reserves. The highest grade beds are contained in the Albert Mines zone, with oil yields averaging 20 gal/ton in the high grade areas. The Albert Mines zone contains an estimated 42.8×10^6 cubic meters (270 million barrels) in situ shale oil reserves in an area 2.6 km^2 to a depth of 600 m (1). These reserves, although appreciable, are modest when compared to very large reserves such as the Green River Formation with an estimated recoverable oil reserve of 680 billion barrels, considering only high grade shale of 25 gallon/ton or higher (2).

The viability of N.B. oil shale would improve if it were utilized with other fossil fuel resources such as high sulphur New Brunswick coal. For this reason one of the concepts under investigation is the possibility of adding a retort to the circulating fluidized bed unit of N.B. Power, located at Chatham, New Brunswick. In this concept the shale is to be retorted followed by injection of the spent shale into the CFB unit. This would optimize resource utilization and could affect environmental and economic advantages. Previous test work at RPC has shown spent shale to be an effective sorbent for capturing sulphur from burning high sulphur coals (3).

2.0 TEST FACILITY

The Research and Productivity Council's pilot scale retort system is shown schematically in Figure 1. It consists of a cylindrical retort, solid feeding and withdrawing equipment, product oil recovery system and gas recycling network. The system components are described below:

2.1 Retort

The cylindrical retort has an inside and outside diameter of 25.4 cm and 61 cm respectively and a total height of 3.5 m. The wall consists of three layers of material; an inner layer of castable high temperature refractory, a middle layer of castable insulating refractory and a carbon steel shell. The thickness of the high temperature and insulating refractory are 5.1 cm and 11.7 cm, respectively.

The cleaned retort off-gases are recycled to the retort as bottom and middle recycle gas. The middle recycle gas, after splitting into two streams, is introduced at a height of 1.4 and 1.85 m measured from the bottom of the retort.

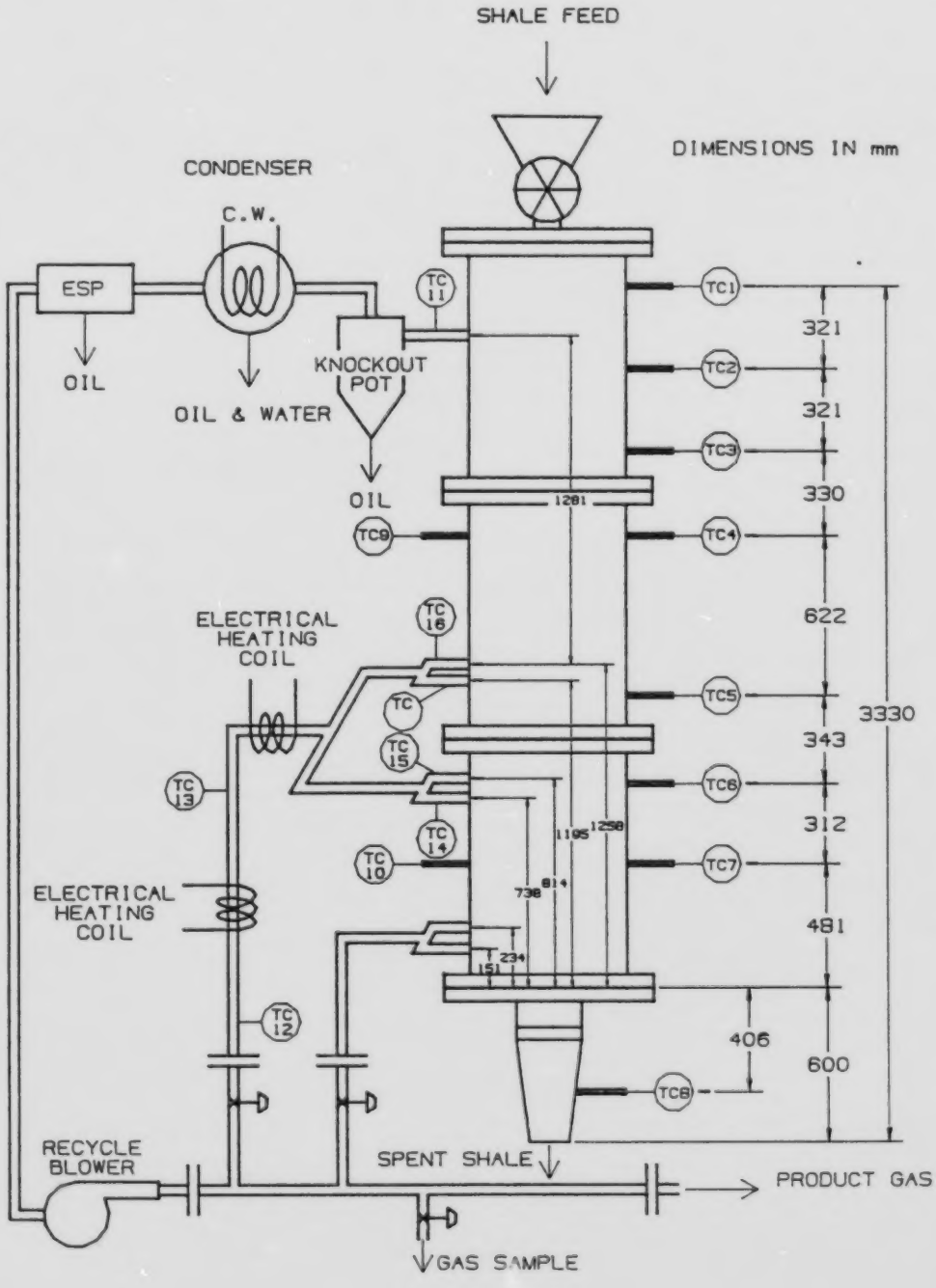
The oil mist-laden off gases are withdrawn near the top of the retort through a side port.

2.2 Solid Feeding and Withdrawal System

Oil shale is fed into the retort by gravity from an overhead hopper through a rotary valve. A predetermined amount of oil shale is loaded into the bin every hour and a timer is used to control the ON/OFF cycles of the rotary valve in order to maintain a steady supply of fresh shale.

FIGURE 1

SCHEMATIC OF RPC FIXED BED RETORT SHOWING INSTRUMENTATION FOR TEMPERATURE AND FLOW MEASUREMENTS



Spent shale is discharged from a conical bin attached to the bottom of the retort. It is fitted with a 6" star-valve equipped with a variable speed drive. The spent shale is charged into a 45 gallon drum mounted on a weighing scale for monitoring and adjustment of the discharge rate in order to maintain a steady shale bed height in the retort. The spent discharge mechanism is shown schematically in Figure 2. This arrangement replaces an earlier screw type design. The new design has operated trouble-free and has led to a substantial improvement in the system's reliability.

2.3 Product Oil Recovery System

Off-gas containing oil mist from the retort undergoes three stages of separation for product oil recovery. These are:

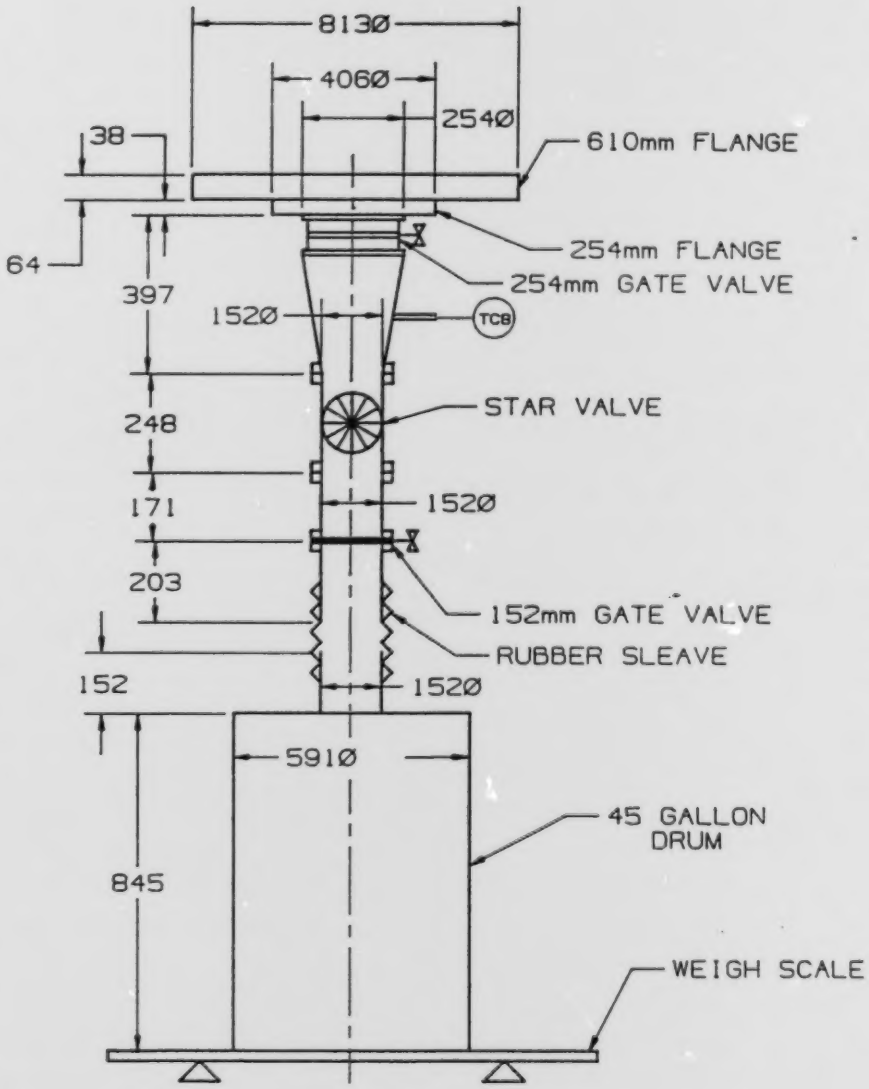
a) a hot knock-out for removing heavy oils and entrained fine particles.
b) two condenser columns with special packing, with an internal column diameter of 0.15 m and an effective height of 4.7 m.
c) an electrostatic precipitator (0.55 m wide x 1.52 m long x 0.13 m thick) with five sets of capacitor cells and ionizers.

2.4 Gas Recycling Network

The cleaned off-gases from the oil recovery system are metered and recycled back to the retort. A portion can be withdrawn as a product gas. The middle recycle is preheated in an electrical heater prior to injection to the two levels of gas feed in the retort. At each level, the preheated gas is distributed using two one inch 316 SS pipes placed at 90° with respect to each other. Each pipe contains 10 holes of 2.4 mm diameter.

FIGURE 2

SCHEMATIC OF SPENT SHALE DISCHARGE MECHANISM



DIMENSIONS IN MM

2.5 Gas Sampling and Analysis

The system is equipped with on-line gas analyzers for CO, CO_2, and O_2 which are used to monitor the composition of retort gas. In addition, a gas chromatograph is also available for batch gas analysis for H_2, N_2 and hydrocarbon content.

3.0 START-UP, TEST AND MEASUREMENTS PROCEDURES

A propane burner was used to preheat the retort. The burner was inserted through a side port located close to the base of the retort. When the temperature at the top of the retort was approximately 800°C, the burner was removed and the side port was sealed. The retort was immediately filled with oil shale and the off-gases were passed through the cleaning train, which consists of a knockout pot, a condenser and an electrostatic precipitator and recycled back into the retort via the electric heater.

The retort was started on shale from RPC's stock-pile in order to conserve the test shales which were in limited supply. The time required to achieve stable operating conditions was approximately 24 hours. The two test shales were then retorted sequentially, each run lasting 24 hours. During each test, operating conditions, such as the flowrate of all input and output streams, retort temperature at several locations, oil and water yields, and on-line gas analysis were recorded on an hourly interval. A record of the raw data is given in Appendix B. Samples of shale oil, retort water and spent shale were collected every 4 hours. Chromotographic analysis of the product gas were carried out every 4 hours.

4.0 RESULTS AND DISCUSSION

The scope of the work as defined in the contract was to characterize two shale samples, carry out retorting tests and characterize the retorting products.

4.1 Raw Shale, Preparation and Characterization

The two bulk samples received represent the Albert Mines Zone (high grade) and the Dolomite Marlstone Zone (lower grade) oil shales from the Canadian Occidental Petroleum Ltd. lease at Albert Mines in southeastern, New Brunswick. The physical appearance of the two shales are quite different. The Albert Mines shale is microlaminated and fissile while the dolomite marlstone zone shale is more massive and competent and less thinly laminated. The samples as received were oven dried at a temperature of 80°C to a moisture content of less than 1%. The two samples were then crushed and screened to the size range of -5/8 inch + 1/8 inch. Each shale was then blended and representative samples were taken for analysis.

The chemical analysis and Fischer assay were carried out and the results are shown in Table 1. Particle size of the prepared feed is given in Table 2.

The Albert Mines sample used in this test contained 32.4 Imp. Gallon/ton. In all the previous work carried out at RPC, this sample represents the highest oil yeild yet recorded. Most previous samples from the Albert Mines had an oil content in the vicinity of 20 Imp. gallon/ton. This sample has also very high calcium content which can be used to capture sulphur from the burning of high sulphur coals.

TABLE 1

CHEMICAL AND PHYSICAL PROPERTIES OF THE TWO SHALE SAMPLES

Determination	ALBERT		DOLOMITE
	Current	Previous*	
Moisture, %	0.62	0.98	0.61
Carbon, %	20.61	11.90	10.51
Hydrogen, %	2.70	1.8	1.49
Nitrogen, %	0.50	0.95	0.47
Sulphur, %	0.94	0.03	0.90
Loss on ignition, %	27.67		18.52
Ash analysis, %:			
SiO_2	44.12	43.97	48.28
Al_2O_3	7.98	12.88	11.98
CaO	11.22	3.80	5.26
Fe_2O_3	3.48	5.44	5.49
K_2O	1.10		1.90
MgO	2.18	1.30	2.65
MnO	0.07		0.10
Na_2O	1.16		1.61
Fischer Assay:			
Oil yield, %	14.2	7.9	4.8
Water yield, %	1.2	3.5	1.5
Gas lost, %	1.9	1.1	1.4
Oil density, g/ml @ 15.5°C	0.8729	0.8738	0.8725
Oil yield Imp. gal/ton (Short)	32.4	16.9	10.9
l/ton (metric)	162.4	84.68	54.6
Heating valve, KJ/kg	7999.0	5582.5	3685.2
Bulk density, g/cm^3	1.21		1.36
Wear **, %	16.6		17.8

* Sample used in the most recent test Program (3).
** Los Angeles Abrasion Test

TABLE 2

PARTICLE SIZE DISTRIBUTION OF THE PREPARED SHALE SAMPLES (WT%)

	ALBERT	DOLOMITE
+ 5/8"	0	0
+ 1/2"	28.3	12.7
+ 4 mesh	66.3	59.5
+ 7 mesh	3.6	16.4
+ 10 mesh	0.4	6.1
+ 45 mesh	0.3	4.3
+ 100 mesh	0.1	0.2
+ 200 mesh	0.1	0.3
- 200 mesh	0.7	0.7

Different and thermal analysis is one in which the heat effects, associated with chemical and physical changes of substance are recorded as a function of temperature (or time) as the substance is heated at a linear rate. The thermal effects may be either endothermic or exothermic, are caused by such physical phenomena as fusion, crystalline structure inversions, vaporization, boiling, sublimation, destructive of crystal lattice and others. Still other enthalpic effects are caused by chemical reactions such as dissociation or decomposition, dehydration, oxidation and reduction, direct combustion, displacement, and so forth. The thermal effects associated with the proceeding physical and chemical changes are measured by a differential method in that the sample temperature is continuously compared against the temperature of thermally inert reference material such as aluminum oxide.

Differential thermal analysis were carried out on the two shales and the results are shown in Figures 3 and 4. The heating rate for all tests was 10°C/min and each shale was tested in air and in nitrogen atmosphere. The Albert Mines shale (A-lot) DTA results are shown in Figure 3. In air atmosphere, physical and chemical changes starts to accure at 683 K (410°C). The large negative exothermic peak is due to the oxidation of the product oil and gases. This process is completed at a temperature of 813°C and the sample temperature does not deviate significantly from the reference inert material. In the nitrogen atmosphere (lower curve), physical changes starts to accure at 610 K, and accelerate rapidly at approximately 730 K. The large endothermic peak continue to the end of the recorded data at approximately 900 K. At this temperature range the shale will be undergong retorting with the associated endothermic physical changes.

The results for the Dolomite Zone shale (D-lot) are shown in Figure 4. In air atmosphere the results are very similar to the Albert Mines shale with an exothermic peak starting at 690 K and completed at 800 K. The size of the peak, however, is smaller than that for the Albert Mines shale. The estimated heat of reaction for the Albert Mines shale and the Dolomite Zone shale are 528 and 321 Cal/g, respectively.

FIGURE 3

DTA CURVES FOR ALBERT MINES SHALE IN AIR AND NITROGEN ATMOSPHERE

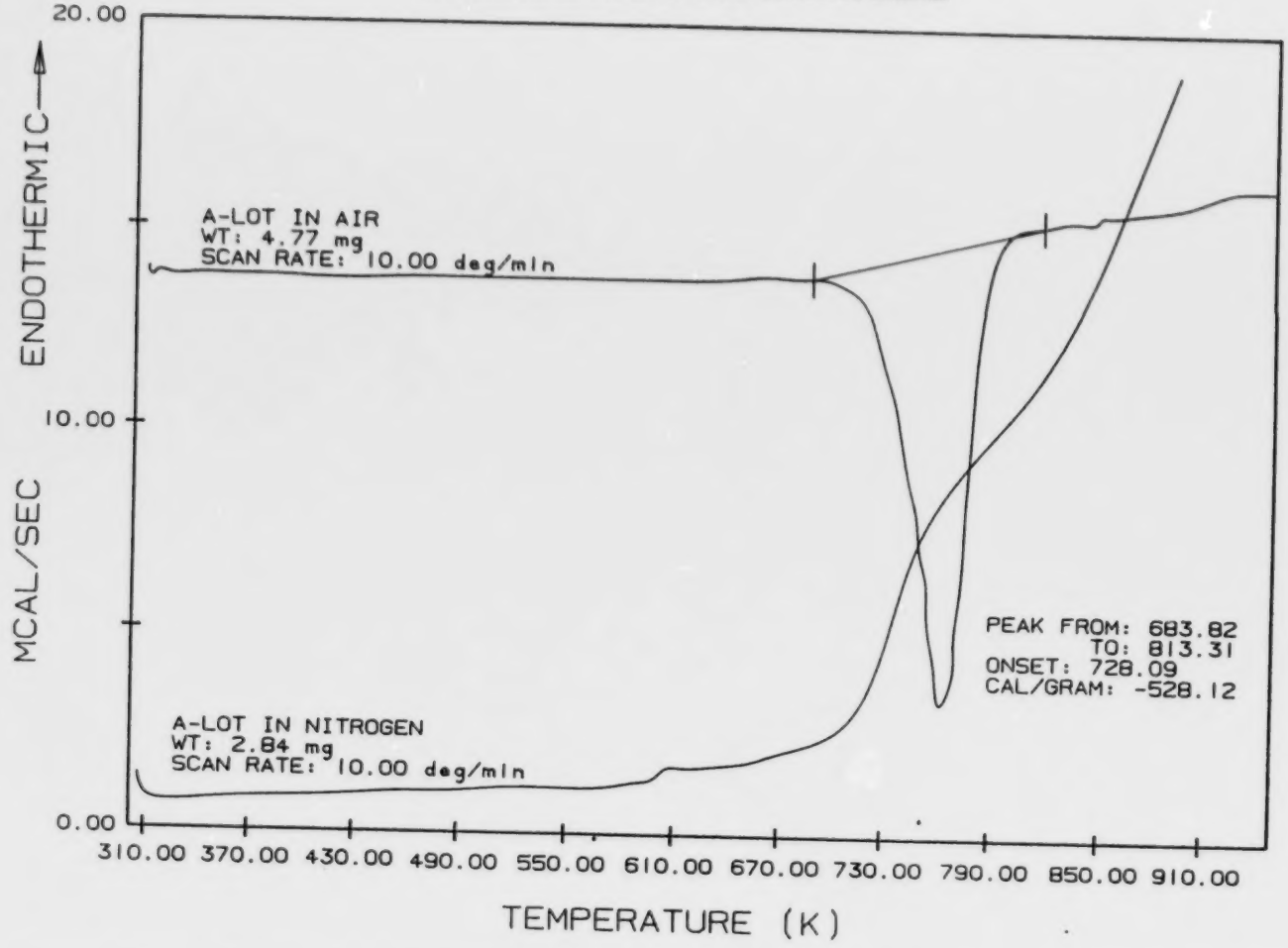
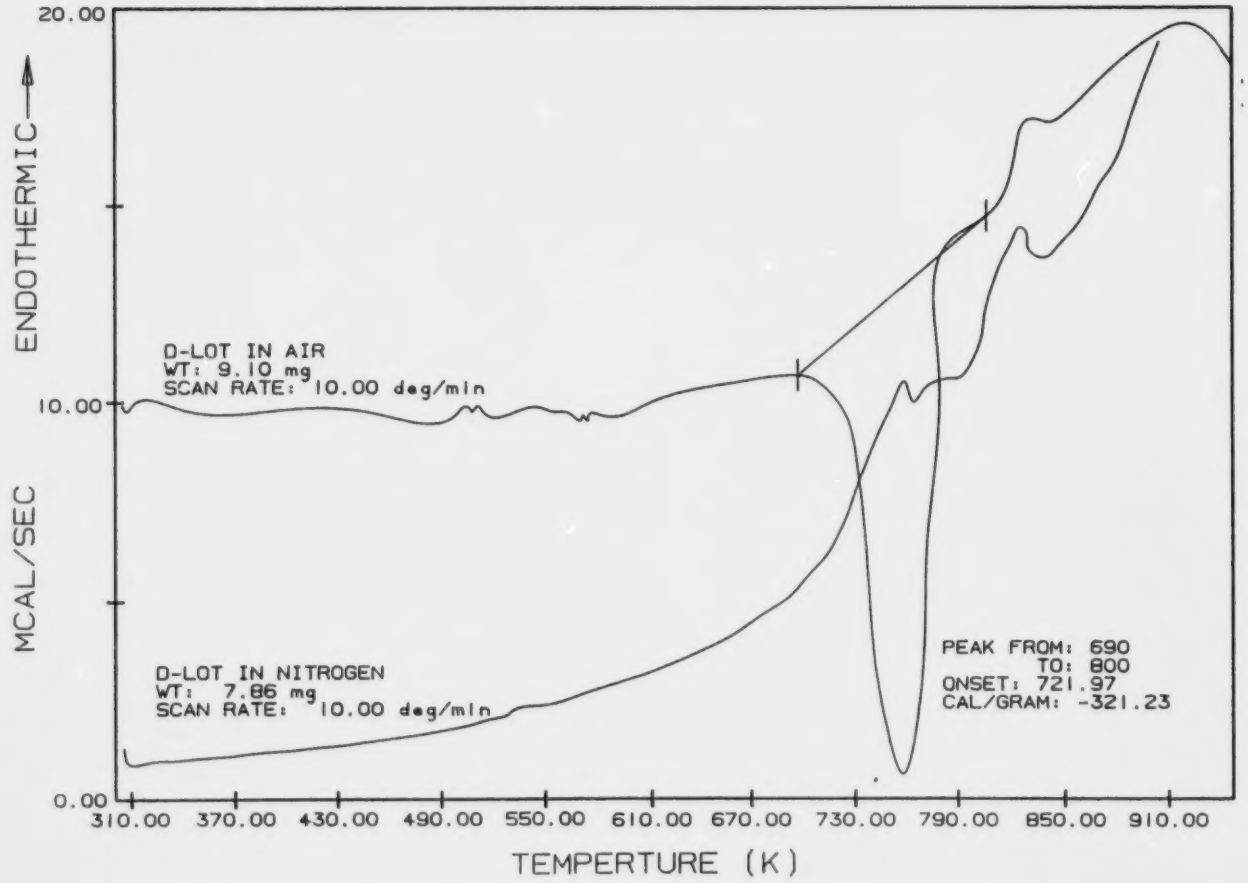


FIGURE 4

DTA CURVES FOR DOLOMITE ZONE SHALE IN AIR AND NITROGEN ATMOSPHERE



At the end of the extothermic peak the curver continues to rise indicating endothermic reactions which could be associated with decomposition of calcium and magnesium carbonets.

In nitrogen atmosphere the Dolomite Zone shale undergoes endothermic physical charge at a lower temperature 490 K as compared to 610 K for the Albert Mines shales. Also there are a number of small peaks which were not identified in the case of the Albert Mines shale.

DTA curves are empirical in nature, therefore, any attempt at quantitative results will require a great deal of attention to experimental conditions such as heating rate, sample size, sample packing, and particle size.

4.2 Operating Conditions and Test Results

Upon completion of the start-up using the procedure described in Section 3, two test runs each of 24 hours duration were carried out sequentially. During the tests, the system operated trouble free and the raw data, recorded on an hourly basis, are given in Appendix B. A summary of the experimental conditions and results are shown in Table 3. Note, the high oil recoveries particularly for the Dolomite Marlstone Zone shale.

The retort temperature profiles are shown in Figures 5 and 6. On each plot the eight points shown are the average temperature from thermocouples 1 to 8 (see Figure 1). The two most important features of the temperature profiles are the width of the retorting zone (i.e. the zone where temerature exceeds 400°C) and the high temperature at the middle of the retort. Both profiles shown in Figures 3 and 4 have a wide retorting zone spanning approximately 50% of the retort height and both shown a high peak temperature (>550°C) at the middle of the retort.

TABLE 3

SUMMARY OF OPERATING CONDITIONS AND RESULTS

	ALBERT	DOLOMITE
Shale feed rate, Kg/hr	20	20
Average bed height, m	2.59	2.69
Shale residence time, hr	7.47	8.72
Net product gas, Kg/hr	2.36	2.27
Total recycle gas Kg/hr	58.8	55.4
Recycled gas to middle, %	74	74
Recycled gas to bottom, %	26	26
Temperature of middle recycle, °C	581	596
Air feed rate, Kg/hr	2.22	1.98
Oil recovery, % of Fisher assay	101.2	134.5

FIGURE 5

DOLOMITE ZONE SHALE
Retort Temperature Profile

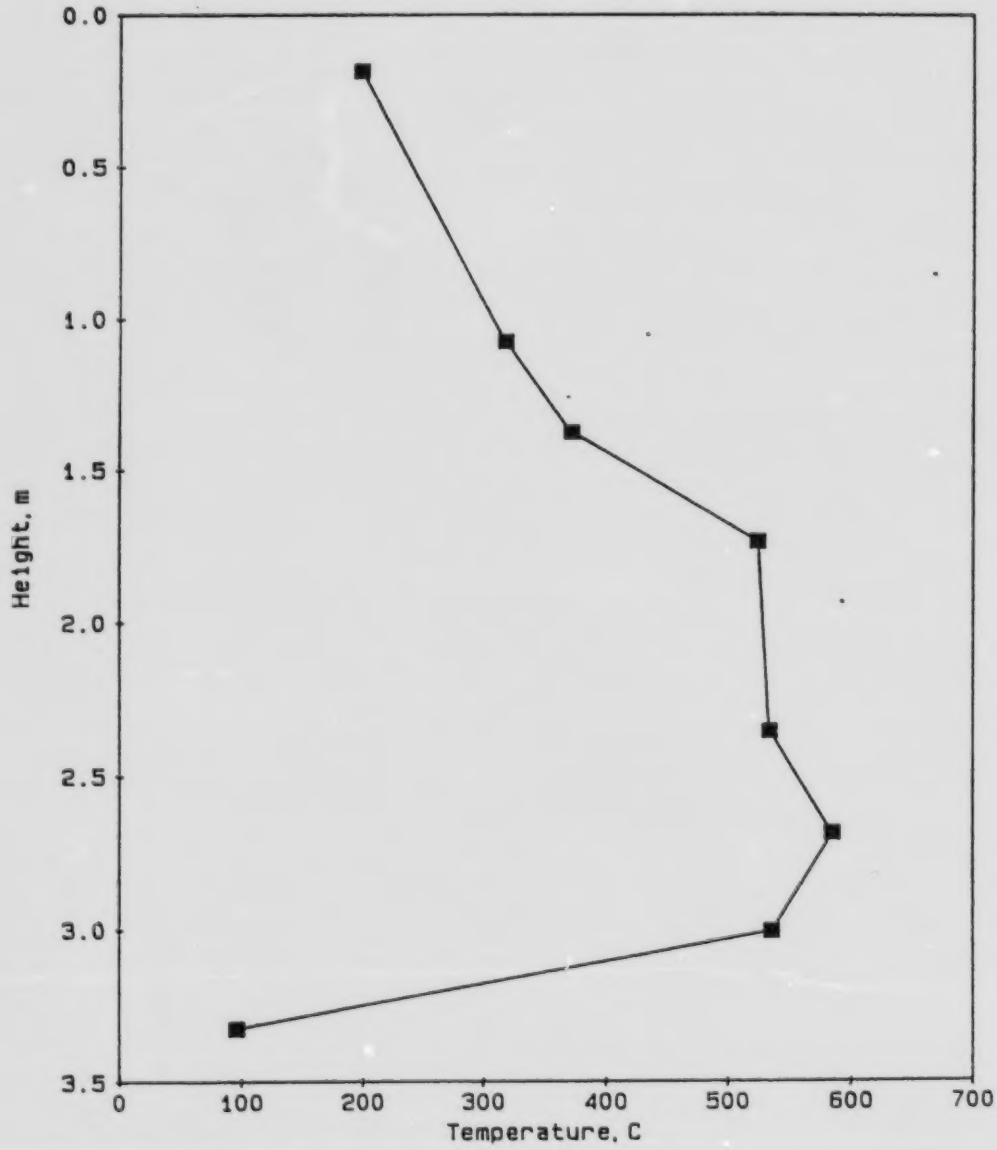
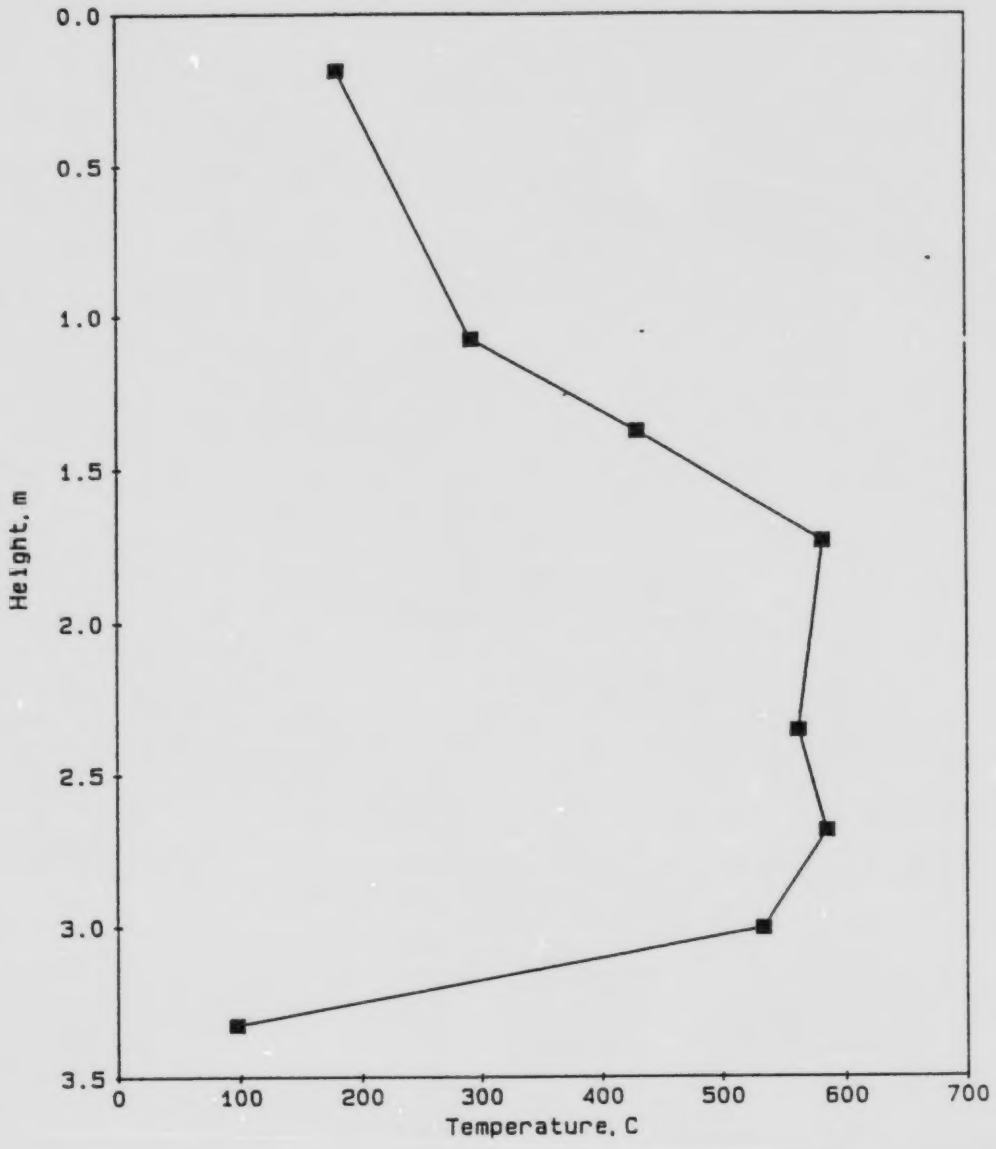


FIGURE 6

ALBERT MINES SHALE
Retort Temperature Profile



Mass and energy balances were carried out on the retort and the results are summarized in Table 4. The errors in the mass balance were small, below 2%, indicating accurate monitoring and control of the input and output streams. Skin losses accounted for 20% of the total heat input to the retort. This is good for pilot scale equipment with high surface area to volume ratio. On scale-up the skin losses should decrease significantly according to the square/cube law. (Retort surface area is proportional to d^2 while its value is proportional to d^3).

The residual heat content in the spent shale represent 8 and 18.7% of the heat content in the fresh shale, for the Albert Mines and Dolomite Marlstone Zone shales, respectively (calculated from Tables 1 and 11). The heating value of the two spent shales shown in Section 4.3.3, are very similar, however, on a percentage basis the heat losses with the spent Dolomite shale are higher than that for the higher grade Albert Mines shale (Table 11).

For the same reasons given above the retort energy efficiency for the Albert Mines shale is higher than the Dolomite Zone shale as shown in Table 5. The retort energy efficiency takes into account the heat content of the shale and external power used to produce a useful form of energy, which in this case is the oil product and net product gas. The result for the Albert Mines shale is good and the retort energy efficiency would improve on scale-up because of lower percentage of skin losses. Commercial size units operating in the indirect retorting mode have an energy efficiency in the vicinity of 75% (9).

Carbon balance was also carried out and the results are summarized in Table 6. The percentage error was small at $\pm$ 7%.

TABLE 4

RETORT MASS AND ENERGY BALANCES

	ALBERT		DOLOMITE	
	Material Balance kg/hr	Heat Balance KJ/kg	Material Balance kg/hr	Heat Balance KJ/kg
Input Streams:				
Raw Shale (21°C)	20	352,757.2	20	162,517.7
Recycle gas to the bottom (63.3, 60.5°C)*	15.05	2,272.1	14.55	2,183.3
Recycle gas to the middle (581.1, 595.6°C)	43.9	74,205	40.95	77,236.1
Air (21°C)	2.23	—	1.95	—
Total In	81.18	429,234.3	77.45	241,941.1
Output Streams:				
Oil (204.7, 236.2°C)	2.95	285,887.7	1.32	127,991.1
Water (204.7, 236.2°C)	0.68	540.1	0.82	756.6
Total off-gas	62.1	33,087.9	57.68	35,415.9
Spent shale (96.5, 95.6°C)	15.95	28,427.2	16.45	30,380.4
Skin heat loss & unaccounted	0.5	81,291.4	1.18	47,398.1
Total out	81.18	429,234.3	77.45	241,942.1
Net product gas **	2.36	9,696.1	2.27	12,897.1

* Stream temperatures for Albert and Dolomite, respectively.
** This represents the fraction of the total produced gas vented to the stack.

TABLE 5

RETORT ENERGY EFFICIENCY

	ALBERT	DOLOMITE
Input:		
Kerogen, KJ/hr	159,710.5	73,579.7
Power, Btu/hr	29,027	30,530
Total, Btu/hr	190,304.5	105,758.3
Output:		
Oil, Btu/hr	129,435	57,942.6
Gas, Btu/hr	4,389.9	5,834.2
Total, Btu/hr	133,824.9	63,781.8
Efficiency:		
Output/Input, %	70.3	60.3

TABLE 6

CARBON BALANCE

	ALBERT	DOLOMITE
Input, kg/hr:		
Shale	4.13	2.10
Output, kg/hr:		
Oil	2.54	1.13
Spent Shale	1.13	0.94
Gas	0.17	0.16
Total	3.84	2.23
Unaccounted	0.29	-0.13
% Error *	+7.0	-6.2

* $\%\ \text{Error} = \frac{\text{In - Out}}{\text{In}} \times 100$

4.3 Retorting Product Characterization

All retorting product streams were sampled every 4 hours. Three samples were selected for chemical analysis for the Albert Mines shale. The samples were taken at the start of the 12th, 16th and 20th hours, while for the Dolomite shale those selected were taken at the 7th, 15th and 23rd hours. Chemical analyses were performed on the individual samples as well as on composite samples made of the three samples for the particular shale.

4.3.1 Product Oils

The product oil is removed from the gas stream in the cleaning train, which consists of a knockout pot, a condenser and an electrostatic precipitator (ESP) and it collects the majority of the oil. Small amounts of oil also are collected in three drain pots located within the pipe network used for gas recycling. Oil from the three drain pots was collected periodically and added to the ESP stream. In the raw data shown in Appendix B this is marked as "other oil". The composite oil samples are made of 9 different oils (three samples from each of the condenser, electrostatic precipitator, and knockout pot).

For each shale, a composite sample as well as an electrostatic precipitator, condenser and knockout pot sample were analyzed and the results are shown in Table 7. Knockout pot oils represent the heavy fractions with an API of -12.3 and -26.3 for the Albert and the Dolomite shale oils, respectively. The two oils also have low carbon contents indicating a high concentration of ash resulting from dust contamination. The knockout pot fractions represent less then 10% of the total and consequently the properties of the composite samples closely resemble the electrostatic and condenser oils.

TABLE 7

SHALE OIL ANALYSIS

Analysis	ALBERT Comp.	ALBERT ESP	ALBERT Cond.	ALBERT Knock out Pot	DOLOMITE Comp.	DOLOMITE ESP	DOLOMITE Cond.	DOLOMITE Knock out Pot
A.P.I.	23.91	24.65	25.10	-12.29	22.67	22.98	23.86	-26.30
Specific Gravity @ 15.5°C	0.9105	0.9062	0.9036	1.1870	0.9178	0.9160	0.9108	1.3451
Viscosity Cst @ 40°C	27.33	24.20	19.21	Solid	30.18	32.46	19.52	Solid
Cst @ 100°C	4.48	4.48	>B.P.*	105.66	4.59	4.76	>B.P.	I/S**
Pour Port, C°	18.3	15.5	10.0	29.4	10.0	12.8	I/S	I/S
Solid, %	88.13	I/S	I/S	I/S	87.58	I/S	I/S	I/S
Carbon, %	85.98	85.44	85.86	60.35	85.76	85.17	85.36	65.26
Hydrogen, %	11.47	11.67	12.07	7.60	11.57	11.62	11.61	7.98
H/C Ratio	1.60	1.64	1.69	1.51	1.62	1.64	1.63	1.47
Nitrogen, %	1.13	1.17	1.09	1.24	1.31	1.06	1.07	1.54
Sulphur, %	0.70	0.63	0.61	1.03	0.56	0.47	0.56	1.46
Heating Value, KJ/kg	42,500	43,531	42,004	30,047	42,370	43,236	42,861	19,132

* Above Boiling Point
** Insufficient Sample

The elemental analyses of the Albert and Dolomite composite oil samples are shown in Table 8. Also show are results from previous test work carried out at RPC on a shale sample taken from Albert Mines, Green River shale oil, Athabasca bitumen and Saudi crude. The elemental analyses of the Albert and Dolomite shale oils are similar with only small variation in the nitrogen and sulphur contents. These oils are also similar in composition to those obtained in the earlier test program

Nitrogen, oxygen and sulphur, known as the heteroatoms are highly poisioness to refining and upgrading catalysts. The concentration of those elements are higher in the Green River formation oil than N.B. shale oil. Nitrogen which is the most undesirable of the hetroatoms is found in much higher concentration in Green River shale as compared to N.B. shale oil.

New Brunswick shale oil, like all other shale oils, contains higher amounts of heteratoms and is difficient in hydrogen when compared to conventional crude, such as Sandi Arabian (Table 8). N.B. shale oil contains less sulphur than Sandi crude, however. The heteratoms must be reduced by hydrotreating prior to treatment of the oil in a conventional refinery.

The physical properties of the product oils are shown in Table 9. Also shown are the physical characteristics of oils from previous test work at RPC on Green River shale oil and Kuwait export crude. The API gravity of the product oils from the present study is slightly lower than the earlier results, which could be due to the fact the oil recoveries in this study were much higher than the previous results (101.2% for the Albert Mines shale as compared to 88.6% for the previous indirect retorting mode results). Nevertheless, N.B. shale oil is still lighter than the Green River shale oil. A pour point of 18.3°C and 10°C is lower than that of Green River oil, but is much higher than the Kuwait crude. A high pour point is an indication of the presence of wax and the paraffinic nature of the crude.

TABLE 8

ELEMENTAL ANALYSIS OF NEW BRUNSWICK AND GREEN RIVER FORMATION SHALE OILS, NATURAL CRUDE AND BITUMEN

Comp.	Currcat Test Results Albert	Dolomitc	NB Oil Shalc * (4&5) Run 13 Indirect	Run 47 Direct	Grecn River Shalc Oil by the Parrahо Process (6)	Athabasca Bitumca (7)	Saudi Arabian Crudc (8)
C	85.98	85.76	85.11 ± 0.04	84.95 ± 0.13	84.5	___	85.00
H	11.47	11.57	11.89 ± 0.03	11.86 ± 0.04	11.20	10.4	13.10
N	1.13	1.31	1.0 ± 0.04	1.02 ± 0.03	1.96	0.60	0.10
O	____	____	0.77 ± 0.02	0.75 ± 0.13	1.60	___	0.10
S	0.70	0.56	0.59	0.56	0.64	3.8	1.70
Ash	____	____	0.64 ± 0.06	0.86 ± 0.03	0.10	0.70	.001
H/C Ratio	1.6	1.62	1.68	1.68	1.59	___	1.85

* Previous Indirect and Direct Retorting test work carried out at RPC, using an Albert Mines shale sample (4 & 5).

TABLE 9

PHYSICAL PROPERTIES OF SHALE OIL *

Properties	N.B. Oil Shale Albert	N.B. Oil Shale Dolomite	N.B. Oil Shale Ind.	N.B. Oil Shale Dir.	Green River Shale Oil (National Research Council, 1980)	Kuwait Export Crude (British Petroleum, 1970)
Gravity, API	23.9	22.7	25.4	25.3	19.8	31.3
Viscosity, Cm^2/s						
20°C	—	—	172	176	—	17
40°C	27.3	30.2	13.4 (38°C)	17.5 (38°C)	24.84 (50°C)	9.6
Pour Point, °C	18.3	10	21.0	15.0	24.0	-25

* Reproduced from Reference (1).

ASTM distillation was performed on Albert Mines and Dolomite Zone shale oil composite samples. The results are given in Appendix C and also shown in Figures 7 and 8. The boiling characteristics of the two shale oils show only minor differences. For example, although the final boiling point of the two oils was not determined, 73.4% of the Dolomite Zone shale oil distilledat 533°C, while the corresponding values for the Albert Mines oil were 69.8% at 536°C. For both oils only 5-6% boiled below 209°C the gasoline range (37-209°C), however, in the jet fuel range (140-310°C) 25-26% distilled off.

The boiling characteristics of the two samples are plotted with those obtained from earlier test work carried out at RPC and with other synthetic and natural crudes as shown in Figure 9 which was reproduced from reference 10. New Brunswick shale oils from this study and those from an earlier study, curves 8 and 9 and 3 and 4, respectively, have similar boiling characteristics to the Green River shale oil (curve 5) up to 250°C. Beyond this point there are differences in the boiling temperatures of these oils with New Brunswick shale oils boiling at lower temperatures. For New Brunswick shale oils the differences between the present and previous results could be attributed to the fact that the oil recovery in the present study was much higher than was achieved earlier (101.2% for the Albert Mines shale as comapred to 88.6% for the previous indirect retorting mode results) and consequently the oils from the present study have a higher boiling range.

4.3.2 Product Gases

Indirect retorting of oil shales, i.e by use of an external heat source as in these tests, normally produces a high heating value product gas. In a commercial process the gas would probably be used on-site for process heat and steam raising for process and possibly for electric power generation.

FIGURE 7

SIMULATED DISTILLATION ANALYSIS FOR THE COMPOSITE ALBERT SHALE OIL SAMPLE

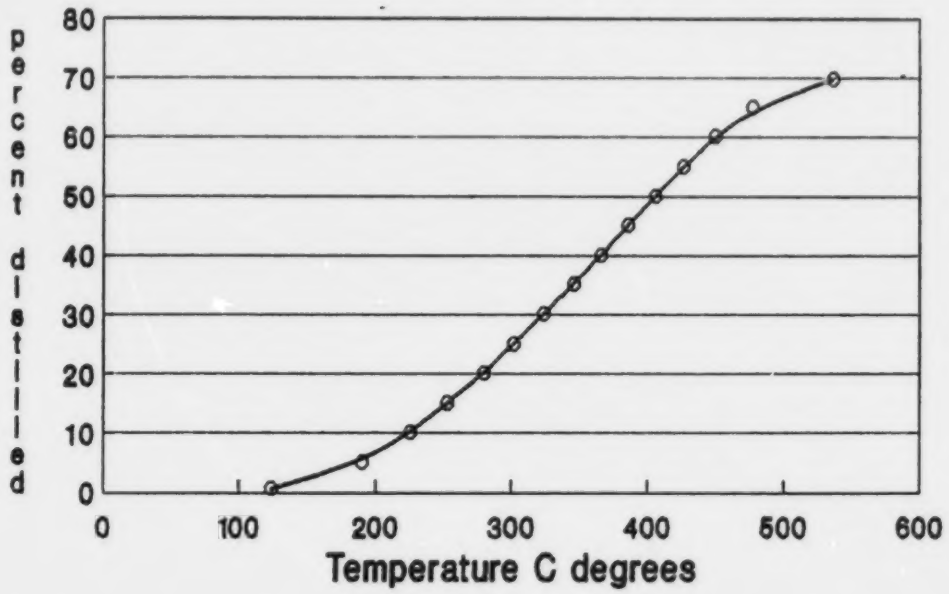


FIGURE 8

SIMULATED DISTILLATION ANALYSIS FOR THE COMPOSITE DOLOMITE ZONE SHALE OIL SAMPLE

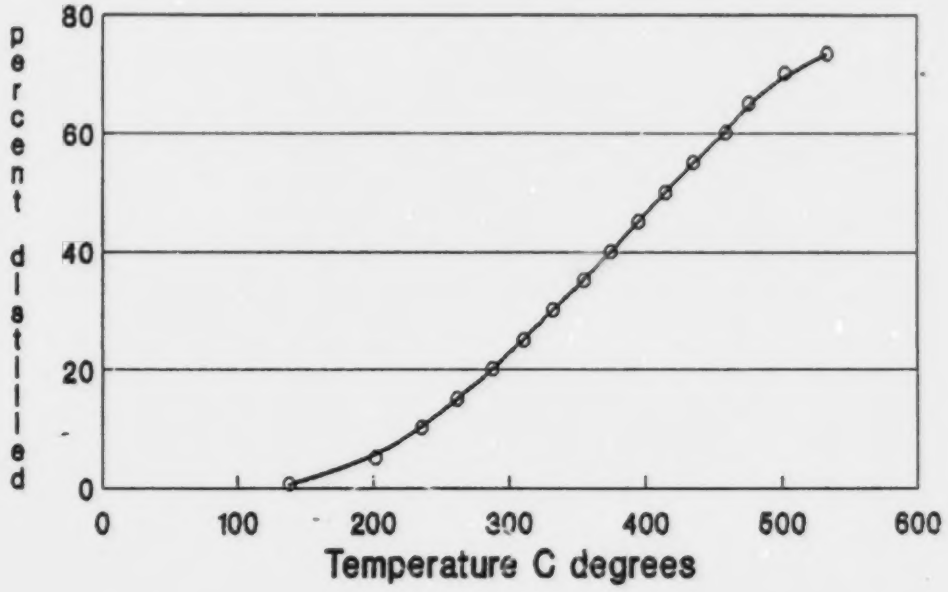
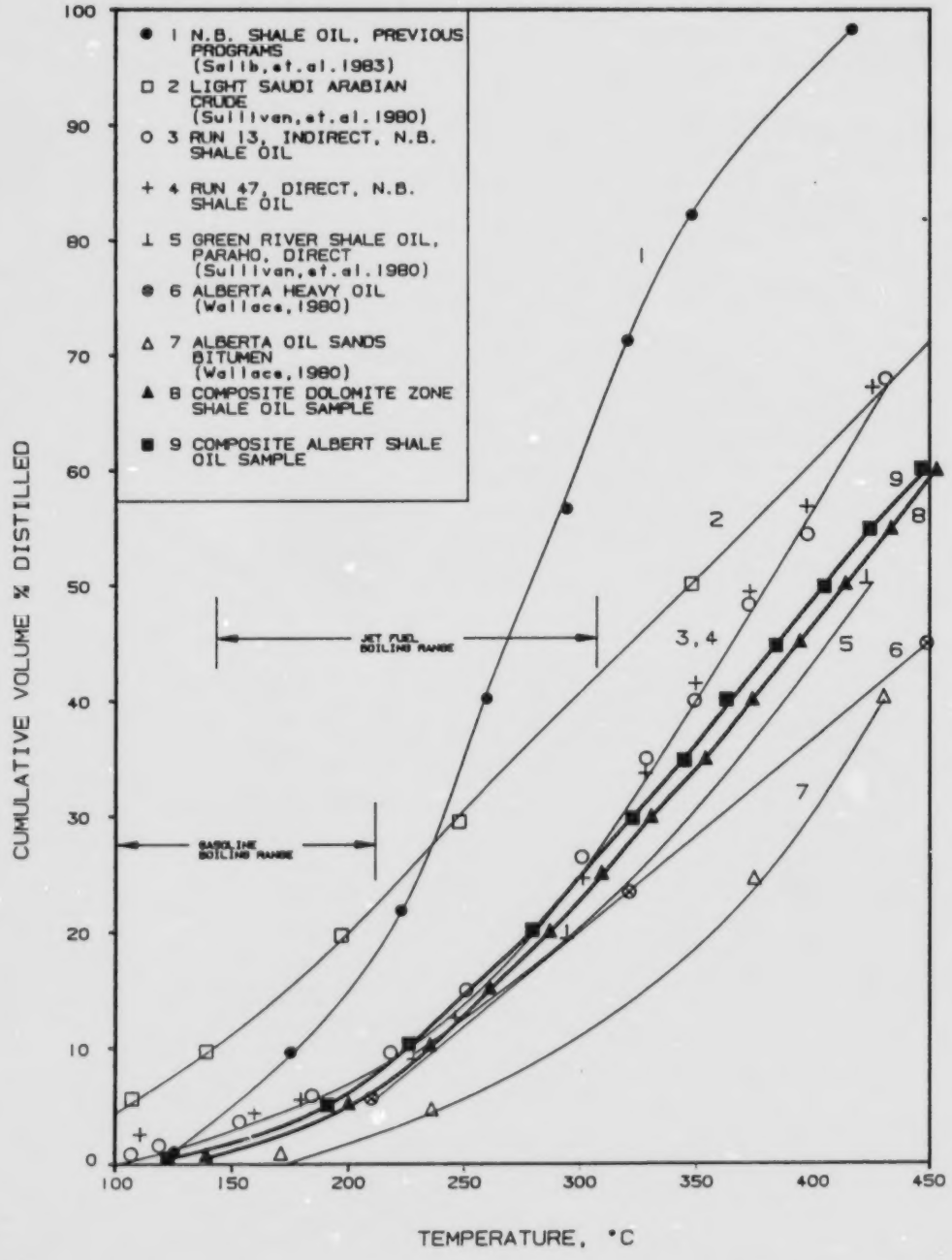


FIGURE 9

DISTILLATION CHARACTERISTIC OF SHALE OILS, HEAVY OIL, OIL SAND BITUMEN AND SAUDI ARABIAN CRUDE



The analyses of the product gases are shown in Table 10. Also shown, is a typical analysis for the Paraho process gas using Colorado oil shale. It is evident from a review of N_2 concentrations, that the product gas produced during the present investigation is diluted with air. The two known sources of air are: (1) that introduced to purge the orifice plates (in order to facilitate gas flow measurments) and (2) infusion while taking measurements of the bed level using a graduated rod which is inserted through an opening at the top of the retort. These two sources of air infusion can be eliminated by mofidication of the existing set-up. The problem, however, would be solved on scale-up using better instrumentation for bed level measurements and control. Also, air purging would constitute a much smaller fraction of total flows.

4.3.3 Retorted Shales

The largest single waste product from oil shale retorting is the spent shale (retorted shale) which must be used or disposed of in an environmently acceptable manner. The chemical analyses of the retorted shales are given in Table 11. The low heating values of the retorted shales is an obivous indication that most of the organic constituents of the shale have been extracted. It also indicate that the remaining carbon is mostly inorganic and is undoubtedly contained within calcium and magnesium carbonates. This is confirmed by the very small amount of oil recovered from the retorted shales by the Fischer assay.

The bulk density of the retorted shales is higher than for the raw shales indicating, in part, that size reduction has taken place (Table 12). The main cause for the increase in density, however, is from the loss of the organic fraction in the spent shale.

TABLE 10

RETORT GAS COMPOSITION (VOL. %)

COMPONENT	ALBERT		DOLOMITE		Parahо Retort Colorado Oil Shale (9)
	Dry	Wet	Dry	Wet	Dry
CH_4	3.1	3.0	3.2	3.1	30.2
CO_2	12.2	11.8	9.9	9.6	14.8
C_2H_6	0.6	0.58	1.9	1.8	5.6
N_2	74	71.8	73	70.8	0.4
CO	0.8	0.78	0.3	0.29	2.7
H_2*	5.0	4.9	5.0	4.9	23.0
H_2O	0	2.7	0	2.7	___
Av. Moleculat wt.	28.27	27.94	27.93	27.37	
Heating Value, KJ/kg	1865		2579		

* Estimated value based on previous test results (3). Hydrogen could not be measured due to a malfunction of the gas chromotograph.

TABLE 11
RETORTED SHALE ANALYSES

ANALYSIS	ALBERT		DOLOMITE	
	Composite *	Sample **	Composite *	Sample **
Moisture, %	0.24	0.34	0.19	0.26
Carbon, %	7.08	7.11	5.70	5.57
Hydrogen, %	0.34	0.31	0.45	0.49
Sulphur, %	0.95	0.72	1.14	1.09
Loss on Ignition, %	14.87	15.04	12.64	12.54
Heating Value, Btu/lb	313	341	326	376
Bulk Density, G/cm^3	2.271	2.292	2.443	2.412
Fischer Assay:				
Weight Loss, %	0.7	1.0	0.6	0.6
Oil Yield, %	0.18	0.28	0.13	0.17
H_2O Yield, %	0.50	0.70	0.40	0.40
Gas Lost, %	0.02	0.02	0.07	0.03
Oil Yield, l/tonne ***	2.1	3.2	1.5	1.9

* Composite is a mixture of 3 samples
** Sample = last individual sample in the particular test
*** Based on an assumed oil density of 0.8725. The actual density could not be measured because the total amount of oil recovered was not sufficient.

TABLE 12

PARTICLE SIZE DISTRIBUTION OF RAW AND SPENT SHALE (WT %)

	ALBERT		DOLOMITE	
	Raw	Spent	Raw	Spent
+ 1/2"	28.3	1.9	12.7	1.4
+ 4 mesh	66.3	41.1	59.5	62.2
+ 7 mesh	3.6	15.9	16.4	12.0
+ 10 mesh	0.4	6.5	6.1	4.4
+ 45 mesh	0.3	17.6	4.3	12.5
+ 100 mesh	0.1	6.4	0.2	2.7
+ 200 mesh	0.1	3.1	0.3	1.4
- 200 mesh	0.7	7.5	0.7	3.2

A comparison of the particle size distribution of the shales both before and after retorting shows that a considerable amount of fines are generated during retorting. The extent of thermal degredation is much greater for the Albert Mines shale than for the Dolomite Marlstone Zone shale. Inspecting Table 12 for the two shales, the following results can be calculated:

	Albert Mines		*Dolomite*	
	Before	**After**	**Before**	**After**
Total -7 mesh	1.6	41.1	11.6	24.2

$$\% \text{ change} \quad \frac{441.1 - 1.6}{41.1} \times 100 = 96.1\%, \qquad \frac{24.2 - 11.6}{24.2} \times 100 = 52.1\%$$

The Albert Mines shale shows an increase in fines of 96.1 whereas the Dolomite ZOne shale shows only half this increase. This is an important finding because a high degree of thermal degredation would decrease the bed premeability and could result in reduction in the reaction rate. In this study no operational problems were encountered, however on scale-up, further work may be required on feed preparation which may include feed blending and tailoring of the feed particle size distribution. Also a freshly mines (unweathered) shale may show different thermal degredation characteristics.

In the literature there are a number of studies on the utilization of spent shales. Potential uses include cement manufacturing and mineral wool production. With regard to disposal, there are a number of options including backfilling the spent shale in the original mined pits. The specific applications an disposal options for N.B. spent shale were outside the scope of this study.

One possible application of N.B. spent shale which has been investigated at RPC is to use the spent shale as a sorbent to capture SO_2 from the burning of high sulphur coal. Previous test work at RPC shows that spent shales are as effective as limestones for SO_2 sorbency. Also, a minimum of 90% of the residual organic carbon in the spent shale can be burnt, thus

producing an ash product with a much reduced potential for environmental contamination.

4.3.4 Process Water

An important consideration in the design of an oil shale retorting facility is treatment and disposal of the water generated in the retorting process. The analyses of the process waters are shown in Table 13. The pH of the process waters is 9.0 and 9.1 for the Albert and Dolomite shale, respectively. These waters contain dissolved minerals and organic materials. The organic material would probably consist of amines, acids, bases and neutral compounds that give the water a dark colour and foul odur. The inorganic components include ammonium, carbonates (bicarbonates), and sulphides (11). The process waters were filtered in the laboratory prior to analysis. Therefore, the suspended solids and the oil and grease contents reported should be considered as lower limits.

By-product recovery from process water is an option. This may include recovery of the phenols which are used in the product of tanning agents, adhesives, and resins. Further work, however, would be required to assess the viability of phenol recovery.

TABLE 13

PROCESS WATER ANALYSES

ANALYSIS	ALBERT	DOLOMITE
Suspended Solids, mg/l	14.6	189.6
Disolved Solids, mg/l	4,590	13,400
Total Solids, mg/l	4,605	13,590
Oil and Grease, mg/l	695	1,000
Phenols, mg/l	19.5	2.7
Sulphide, g/l	1.67	5.00
NH_3, %	3.6	2.5
CO_2, mg/l	<20	<20
pH	9.0	9.1

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The main conclusions of this study are as follows:

1. New Brunswick oil shales from the Albert Mines and the Dolomite Marlstone zones can be retorted efficiently using RPC's pilot scale retort operating in the indirect-heating mode. The oil yields achieved for the Albert and Dolomite shales were 101.2 and 134.5% of the Fischer assay, respectively.

2. For the high grade Albert Mines shale, high thermal energy recovery of 70% was achieved. The corresponding value for the lower grade Dolomite shale was 60%.

3. New Brunswick shale oil is lighter and contains less heteroatoms than the Green River shale oil produced by the Paraho indirect retoring process.

4. New Brunswick shale oil will require hydrotreating before it can be used in a conventional refinery. Upon upgrading, however, it would be a suitable feedstock for the production of gasoline, kerosen, jet and diesel fuels.

5. The heating values of the spent shale from these particular tests were low varying between 729 to 875 KJ/kb. The spent shale from the Albert Mines shale contains a high concentration of calcium which could make it a technically and economically viable sulphur sorbent agent. It could be used to capture sulphur from burning high sulphur coals.

6. Albert Mines shale is shown to be more suseptable to thermal degradation than the Dolomite Marlstone Zone shale. This could have a retarding effect on the retorting rate of the Albert Mines shale and an increased pressure drop in the retort.

5.2 Recommendations for Future Work

Future research and development work is required in order to provide the necessary information required to assess the potentail of an oil shale mining and refining industry in New Brunswick. Further work identified as a result of this study includes:

1. The Albert Mines sample used in these tests was of high grade, yielding 32.4 Imp. Gal/ton (short) (162.4 l/tonne). It also contains a high concentration of calcium (11.2% CaO). The results represent the highest oil grade and the highest calcium concentrations and if the shales are present in large quantities could have a positive impact on the viability of New Brunswick oil shale as an energy source as well as a sulphur capture agent. It would, therefore, be worthwhile to undertake further studies to assess the grade, resources, and properties of Albert County oil shale deposits.

2. Further tests are recommended for the optimization of the retorting process in terms of gas and oil production, retorting rate, product gas, fines generation and energy efficiency. This should be undertaken both on the pilot scale as well as on the demonstration scale (10-15 tons/day).

3. The economics of NB oil shale exploitation should be investigated. This may include a number of scenarios including co-utilization in existing and new coal burning plants.

6.0 ACKNOWLEDGEMENTS

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Special thanks are due to Donald Barnett and Clint St. Peter for their support of the project. The assistance of Ross Gilders and other RPC technical personnel in carrying out the work is gratefully acknowledged.

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8.0

AUTHORIZATION

DEPARTMENT: **MINERALS AND FUEL TECHNOLOGY**

PROJECT TITLE: **"TESTING OF NEW BRUNSWICK OIL SHALE IN RPC TEST FACILITY"**

PROJECT NUMBER: 08008046/0001 NUMBER PROPOSAL MDPP/88/16
REPORT MFT/89/07

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APPENDIX A

CONTRACT

CONTRACT

RPC proposal MDPP/88/016 dated November 7, 1988, describes in detail all the technical tasks to be carried out in fulfilment of the contract. A copy of the proposal is included as a reference.

TESTING OF NEW BRUNSWICK OIL SHALE IN R.P.C. TEST FACILITY

A Proposal Submitted

to

Mr. Donald E. Barnett
Director of the Energy Branch
New Brunswick Department of Natural Resources
P.O. Box 6000
Fredericton, NB
E3B 5H1

Proposal No.: MDPP/88/016
Date: November 7, 1988

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Minerals & Fuel Technology

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APPENDIX A REQUEST FOR PROPOSAL

APPENDIX B RESUMES OF KEY PERSONNEL

1.0 INTRODUCTION

This proposal is submitted in response to the Energy Branch of the N.B. Department of Natural Resources and Energy's request for proposal dated October 21, 1988. The requirement is to evaluate the physical characteristics and shale oil recoveries for two shale samples.

As outlined in the request for proposal which is included in Appendix A, the Department of Natural Resources, N.B. Power, Can Oxy and EMR are interested in the extraction of hydrocarbons from N.B. Oil Shale. This project is a first step in a comprehensive economic evaluation of the concept of integrating an oil shale retort with the Circulating Fluidized Bed Unit of N.B. Power located at Chatham, N.B.

The Research and Productivity Council has been involved in the processing of oil shale since the early 1980's. In 1982 RPC designed, built and operated the only Canadian pilot scale retort. A second retort of a much improved design was commissioned later in 1985. Our circulating fluidized bed unit, at the time of commissioning, was the only operating pilot scale unit in Canada. RPC is therefore well qualified to carry out this project.

The work will be undertaken by the Department of Minerals and Fuel Technology at RPC. The project could be completed within four months of authorization at a total estimated cost of $49,984.00

2.0 METHOD OF APPROACH

2.1 Scope of Work

The scope of the work as defined in the request for proposal is as follows:

a) Characterization of two oil shale samples.
b) Retorting of the two samples using RPC's pilot scale retort and characterization of the retorting products (oil, gas and spent shale).
c) Data analysis and reporting.

The work will be carried out at RPC's existing test facilities which are described below.

2.2 Test Facility

The Research and Productivity Council's pilot scale retort system is shown schematically in Figure 1. It consists of a cylindrical retort, solid feeding and withdrawing equipment, product oil recovery system and gas recycling network. The system components are described below:

Retort

The cylindrical retort has an inside and outside diameter of 25.4 cm and 61 cm respectively and a total height of 2.9 m. The wall consists of three layers of material; an inner layer of castable high temperature refractory, a middle layer of castable insulating refractory and a carbon steel shell. The thickness of the high temperature and insulating refractory are 11.7 cm and 5.1 cm respectively.

The cleaned retort off-gases are recycled to the retort as bottom and middle recycle gas. The middle recycle gas, after splitting into two streams, is introduced at a height of 0.8 and 1.25 m measured from the bottom of the retort.

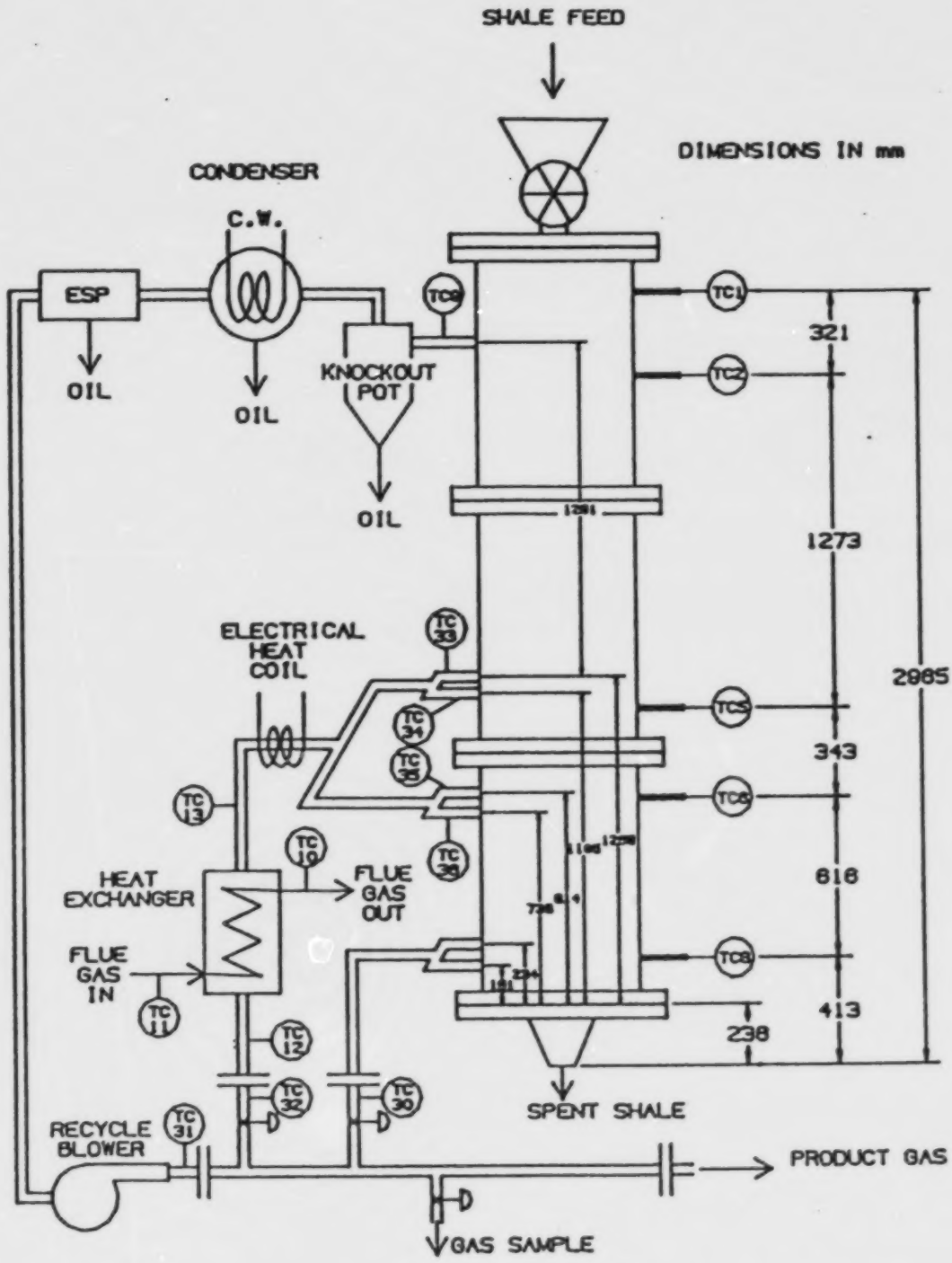


FIGURE 1: SCHEMATIC OF RPC'S PILOT-SCALE RETORT SYSTEM.

The oil mist-laden off gases are withdrawn near the top of the retort through a side port.

Solid Feeding and Withdrawal System

Oil shale is fed into the retort by gravity from an overhead hopper through a rotary valve. A predetermined amount of oil shale is loaded into the bin every hour and a timer is used to control the ON/OFF cycles of the rotary valve in order to maintain a steady supply of fresh shale.

Spent shale is discharged from a bin attached to the bottom of the retort through a variable-speed screw feeder into a receiving pot. The discharge rate is set so as to maintain a steady shale bed height in the retort. Bed level is measured periodically with a graduated rod.

Product Oil Recovery System

Off-gas containing oil mist from the retort undergoes three stages of separation for product oil recovery. These are:

a) a hot knock-out for removing heavy oils and entrained fine particles.
b) two condenser columns with special packing, with an internal column diameter of 0.15 m and an effective height of 4.7 m.
c) an electrostatic precipitator (0.55 m wide x 1.52 m long x 0.13 m thick) with five sets of capacitor cells and ionizers.

Gas Recycling Network

The cleaned off-gases from the oil recovery system are metered and recycled back to the retort. A portion can be withdrawn as a product gas. The middle recycle will be preheated in an electrical heater prior to injection to the two levels of gas feed points in

the retort. At each level, the preheated gas is distributed using two one inch 316 SS pipes placed at 90°C with respect to each other. Each pipe contains 10 holes of 2.4 mm diameter.

Gas Sampling and Analysis

On-line gas analysis for CO, CO_2 and O_2 will be used to monitor the composition of the retort gases. In addition, tubes of retort gases will be collected for subsequent analysis of H_2, N_2 and hydrocarbon content using gas chromatography.

3.0 PROJECT EXECUTION

The project will be subdivided into five main tasks as follows:

3.1 Feed Preparation and Characterization (Task 1)

The two samples will be delivered to RPC in drums. The samples will be dried to a moisture content of approximately 1% using a low temperature (~ 80°C) oven in order to avoid any decomposition. The samples will then be crushed to a top size of 1/2". This will be followed by screening to remove the -1/8" fines.

The total weight of the two samples, as mentioned in the request for proposal, is only 4 tonnes. Depending on the moisture content and the amount of fines in the samples, the useful fraction per sample could be as low as one tonne. This is probably sufficient to complete the evaluation, however, some additional shale from RPC's present stockpile will be prepared and used for the retort start-up. Once this is completed and the retort is fully operational, the two samples will be tested sequentially.

The two shales will be sampled and characterized. The following analyses will be carried out:

- Moisture
- Fischer assay
- Loss on ignition
- Carbon, hydrogen, sulfur
- Heating value
- Inorganic analysis (whole rock analysis)
- Differential thermal analysis
- Specific heat
- Particle specific gravity
- Drop test
- Los Angeles Test
- Bulk density
- Size distribution

3.2 Retort Preparation (Task 2)

System preparation will consist of routine maintenance, some modifications and the design and construction of an electric heater. System maintenance will include replacement of broken thermocouples and heating elements, replacement of condenser packing, cleaning and recalibration of flow meters, etc.

The only major modification necessary is the replacement of the existing spent shale discharge screw with a more reliable device. A star-valve arrangement would improve the system reliability. Most of the necessary hardware is available in our inventory.

In this study the retort will be operated in the indirect mode* i.e. the heat requirement for the retort is supplied from an external source. A special electrical heater will be built for this purpose.

3.3 Test Runs (Task 3)

The retort will be started up using shale from our existing stockpile. Once the system has stabilized and all components are functioning properly, the two test shales will be fed into the retort sequentially. This is intended for the economical use of the test shale samples which are in limited supply.

The duration of each test will be approximately 24 hours, which is sufficient time to change the bed over four times. This will ensure that the results represent steady state operation.

The operating conditions will be selected based on previous experience. In a recent test program using high kerogen shale from Albert County, high oil recoveries were obtained using the following set of operating conditions.

* The Petrosix process operates in the indirect velocity mode.

Shale Feed Rate, kg/hr	18.0 ± 10%
Average Bed height, m	2.4 ± 10%
Shale Residence Time, hr	7.7 ± 10%
Total Recycle Gas/1000kg of Shale	318 ± 10%
Recycle Gas to the Middle, %	78 ± 10%
Recycle Gas to the Bottom, %	22 ± 10%
Temperature of Middle Recycle Before entering the retort, °C	550 ± 10%
Air Feed to the Bottom of the retort, kg/100kg of shale	0-7

During each test, the operating conditions such as: feed rate of the input streams; recycle rates; retort temperature at several locations; oil and water yields; etc., will be recorded on an hourly basis. Samples will be collected of the retort gas, shale oil, retort water and spent shale for subsequent analysis.

3.4 Chemical and Data Analysis (Task 4)

Three samples of each product stream will be analyzed. The analyses to be carried out are as follows:

For the shale oil:

- Density
- Viscosity
- Pour point
- Solids content
- Carbon
- Hydrogen
- Sulphur
- Nitrogen
- Heating value
- ASTM distillation

For the retort gas:

- Chromatographic analysis for hydrocarbons, hydrogen and nitrogen. On line gas analysis will also be carried out for CO, CO_2 and O_2.

For the retort water:

- Dissolved and total solids
- Phenols
- Sulphides
- Nitrogen as NH_3
- Carbon dioxide
- Oil content
- pH

For the retorted shale:

- Moisture
- Fischer assay
- Carbon, hydrogen, sulphur
- Heating value
- Loss on ignition
- Bulk density
- Size distribution

The results of the chemical analyses together with the operating conditions, will provide all the necessary information for mass and energy balances.

3.5 Final Report Preparation (Task 5)

The final report will give a full description of the test facility, test conditions and results and chemical analyses. It will also include discussion, and conclusions on the retorting characteristics of the two shales, together with recommendations for future work.

4.0 MANAGEMENT PROPOSAL

4.1 Management and Personnel

The project will be undertaken by the Department of Minerals and Fuel Technology of the Research and Productivity Council. The overall management of the program will be by P. Salib, Head, Fuel Technology. R. Gilders will provide technical assistance. Other RPC professionals and technicians will be used as necessary. Resumes of key personnel are attached in Appendix B.

4.2 Project Schedule

The project can be completed within four (4) months of commencement.

5.0 PRICE PROPOSAL

The total expenditure anticipated for this project is $49,984.00. The cost per task including labour charges are shown in Table 1. A breakdown of chemical analysis costs and for materials and supplies are given in Tables 2 and 3, respectively.

TABLE 1

SUMMARY OF COSTS

Project tasks and Associated Costs	No. of hours hr	Rate $/hr	Total Cost $
Feed Preparation and Characterization:			
- Casual Labourer	80	10	$ 800.00
- Chemical analysis*	--	--	$ 1,200.00
Sub-Total			$ 2,000.00
Retort Preparation:			
- Casual Labourer	80	10	$ 800.00
- Technician II	80	42	$ 3,360.00
- Professional VII	36	85	$ 3,060.00
- Material and Supplies**	--	--	$ 6,000.00
Sub-Total			$13,220.00
Test Runs:			
- Casual Labourer	64	10	$ 640.00
- Technician I	96	31	$ 2,976.00
- Technician II	96	42	$ 4,032.00
- Professional VII	192	85	$16,320.00
Sub-Total			$23,968.00
Chemical and data analysis:			
- Professional VII	20	85	$ 1,700.00
- Chemical analysis	--	--	$ 4,740.00
Sub-Total			$ 6,440.00

TABLE 1 (CONT'D)

Project tasks and Associated Costs	No. of hours hr	Rate $/hr	Total Cost $
Final report preparation:			
- Professional VII	36	85	$ 3,060.00
- Professional VIII	12	108	$ 1,296.00
Sub-Total			$ 4,356.00
GRAND TOTAL			$49,984.00

* See Table 2

** See Table 3

TABLE 2

ESTIMATED COSTS FOR CHEMICAL ANALYSIS

Item	Cost per Sample $	No. of Samples*	Total Cost $
Raw Oil Shale	$600	2	$1,200
Shale Oil	$250	6	$1,500
Retort Gas	$120	6	$ 720
Retort Water	$150	6	$ 900
Retorted Shale	$270	6	$1,620
TOTAL COST			$5,940

* Two raw oil shale samples and three samples per test of each of the product streams.

TABLE 3

ESTIMATED COSTS FOR MATERIALS AND SUPPLIES

Item	Estimated Cost ($)
Thermocouples	$ 800
Speciality Gases	$ 600
Glasswear	$ 300
Insulation	$ 300
Heating Elements	$ 600
Packing Material	$ 500
Fittings	$ 200
Stainless Steel Pipes	$ 300
Valves	$ 100
Flexible Stainless Steel Hoses	$1,000
Expansion Joint	$ 200
Safety Supplies	$ 300
Electrical Supplies	$ 300
Miscellaneous	$ 500
TOTAL	$6,000

AUTHORIZATION

DEPARTMENT: **MINERALS AND FUEL TECHNOLOGY**

PROJECT TITLE: **"Testing of New Brunswick Oil Shale in R.P.C. Test Facility"**

PROJECT NUMBER: PROPOSAL NUMBER **MDPP/88/016**

REPORT

Prepared By: _____
P. Salib

Reviewed By: _____
Dr. D. Abbott

Approved By: _____
Dr. R.S. Boorman

Date: **November 8, 1988**

rpc RESEARCH AND PRODUCTIVITY COUNCIL
P.O. Box 6000, Fredericton, New Brunswick, Canada, E3B 5H1
Telephone (506) 452-8994
Telex 014-46115
Fax (506) 452-1395

APPENDIX A

REQUEST FOR PROPOSAL

REQUEST FOR PROPOSAL

TO

NEW BRUNSWICK RESEARCH AND PRODUCTIVITY COUNCIL (R.P.C.)

FOR

TESTING OF NEW BRUNSWICK OIL SHALE

IN

R.P.C. TEST RETORT

Energy Branch
New Brunswick Department of Natural Resources and Energy
P.O. Box 6000, Fredericton, New Brunswick E3B 5H1

October 14, 1988

A. **BACKGROUND:**

The Department of Natural Resources and Energy and N.B. Power in cooperation with Canadian Occidental Petroleum Ltd. and Energy, Mines and Resources Canada ~~are~~ is interested in the extraction of hydrocarbons from New Brunswick oil shale. The concept being explored is that of a possible retort addition to the circulating fluidized bed unit of N.B. Power, located at Chatham, New Brunswick. Under this concept it is thought that the extraction of shale oil followed by injection of the spent shale into the CFB unit may optimize resource utilization as well as offer economic advantages.

As a first step in analyzing the economics of the concept it is required that both the high kerogen shale and the dolomitic shale, which occur at Albert Mines, be evaluated as to their physical characterization and shale oil recoveries.

Nine drums of high kerogen and nine drums of dolomitic shale have been collected and will be supplied. Preparation of the shale samples for retort feedstock will be required. Similar samples of both shales have been submitted to Petrobras, Brazil via Stone and Webster Engineering Corporation, who will assist Petrobras in the pilot plant testing and data acquisition.

B. SCOPE OF WORK:

1. Technological Characterization of Raw Oil Shale Samples:

   (a) High Kerogen Shale
   (b) Dolomitic Shale

2. Pilot Plant Tests Including Characterization of Products and Spent Shale:

   (a) High Kerogen Shale
   (b) Dolomitic Shale

Raw shale, product fluids and spent shale testing shall include tests as indicated in Schedule "A".

.../2

C. **REPORT:**

The report of work shall include but not necessarily be limited to:

1. Introduction

2. Pilot Plant Installation Summary:

   (a) Equipment

   (b) Process Flow
   - (i) Solids
   - (ii) Gas
   - (iii) Liquid

   (c) Instrumentation and Control:
   - (i) Retort Feed System
   - (ii) Retorting Rate
   - (iii) Operating Pressure
   - (iv) Recycled Gas Temperature
   - (v) Liquid Handling System
   - (vi) Gas Measuring and Sampling

3. Characteristics of Raw Shale:

   (a) Handling
   (b) Assays and tests
   (c) Discussion of Results

4. Pilot Plant Stage:

   (a) Physical Assays
   (b) Pilot Plant Test Results
   (c) Discussion of Results

5. Conclusions

6. Recommendations

C. **ADMINISTRATION**

1. Proposal should include per diem charges for all staff charged to the project and the number of days each will spend on the project. All overhead charges, operating costs and other installation charges should be clearly indicated.

2. Schedule of work should be given.

.../3

3. The Department of Natural Resources and Energy will be responsible for delivery to R.P.C. of the samples of raw shale. The samples will be in eighteen (18) drums each three feet in height and two feet in diameter. The weight of the eighteen drums will be about 4 tonnes.

4. Payments shall be linked to the schedule and there shall be a ten percent (10%) holdback of funds pending acceptance of the final report.

5. Proposal acceptance is subject to available funding under the Mineral Development Agreement as well as cost effectiveness relative to the parallel Petrabras testing.

6. There shall be no compensation for any or all expenses incurred in preparation of the proposal.

SCHEDULE "A"

SAMPLE TESTS

During the tests, samples will be taken of the raw shale, oil, gas, water and spent shale for determination of the most important properties which follow:

- For the raw oil shale
  - Moisture
  - Fischer assay
  - Loss on ignition
  - Carbon, hydrogen, sulfur
  - Heating value
  - Inorganic analysis (whole rock analysis)
  - Differential thermal analysis
  - Specific heat
  - Particle specific gravity
  - Drop test
  - Los Angeles Test
  - Bulk density
  - Size distribution

- For the shale oil
  - Density
  - Viscosity
  - Pour point
  - Solids content
  - Carbon
  - Hydrogen
  - Sulphur
  - Nitrogen
  - Heating value
  - ASTM distillation

- For the retort gas
  - Chromatographic analysis

- For the retort water
  - Dissolved and total solids
  - Phenols
  - Sulphides
  - Nitrogen as NH_3
  - Carbon dioxide
  - Oil content
  - pH

- For the retorted shale
  - Moisture
  - Fischer assay
  - Carbon, hydrogen, sulphur
  - Heating value
  - Loss on ignition
  - Bulk density
  - Size distribution

APPENDIX B

RESUME OF KEY PROFESSIONALS

RESEARCH AND PRODUCTIVITY COUNCIL

PHILIP F. SALIB B.Sc., M.Sc. Tech., Ph.D., M. InstE., C. Eng., P. Eng.
Head, Fuel Technology
Minerals and Fuel Technology

Education

1978 - 1982	Ph.D., Sheffield University U.K., Chemical Engineering and Fuel Technology
1976 - 1978	M.Sc.Tech., Sheffield University U.K., Chemical Engineering and Fuel Technology
1966 - 1971	B.Sc. Alexandria University, Egypt, Chemical Engineering

Specialties

Coal Combustion, Process Simulation and Oil Shale Technology

Experience

1982 - Present	Head, Fuel Technology
1978 - 1982	Coal Combustion Consultant - Coal Research Establishment U.K.
1972 - 1976	Production Engineer-Cociete Industrial Maharrm Press. Alexandria, Egypt. Responsibilities include Raw Material Control and Energy Monitor, Supervision of Manpower and Scheduling of Maintenance.

Relevant Professional Experience

Formulated and carried out comprehensive program for the co-utilization of high sulphur coal and oil shale.

Designed, constructed and tested a moving bed retort for the production of liquid petroleum fuels from oil shale.

Planned combustion emission control (SOx, NOx) tests with multi-staged burner for pulverized coal and supervised test program for the New Brunswick Electric Power Commission.

Designed and constructed the first operational circulating fluidized bed combustion pilot plant in Canada.

Carried out circulating fluidized bed combustion tests on Canadian coals and limestones to establish combustion efficiency and sulphur capture.

Developed concept, designed and constructed equipment and carried out test programs to show the technical feasibility of producing liquid petroleum products from oil shale using heat from a process integrated with a circulating fluidized bed combustor fired with coal and spent shale. This process reduces sulphur emissions to a minimum.

Associations

Member of the N.B. Association of Professional Engineers

Registered Chartered Engineer (UK)

Member of Institute of Energy

Member of Combustion Institute

Publications

1. **Salib, P., Barua, S., Robson, K. and Kissell, R.,** "Circulating Fluidized Combustion of Different Ranking Coals with Limestone Sorbents". Ninth International Conference, Fluidized Bed Combustion, Boston, May 1987.

2. **Salib, P., Barua, S., Furimsky, E.,** "Retorting of Oil Shales from New Brunswick, Canada", Can. J. Chem. Engl, 64, December 1986.

3. **Salib, P., Barua, S.,** "Pyrolysis of Eastern Canadian Coals and Oil Shales in a Circulating Fluidized Bed Reactor", RPC Report AS/86/16, 1985.

4. **Barua, S., Salib, P.,** "Detailed Characterization of Albert Mines Shales Oil and Its Fractions Produced at RPC", RPC report AS/85/76, 1985.

5. **Salib, P., Sy. O., Barua, S., Gemmell, D. and Furimsky, E.,** "Demonstration of an Integrated Fluidized Bed Combustor and Oil Shale Retort System", presented at the Eastern Oil Shale Symposium, Lexington, Kentucky, November 4, 1984.

6. **Barua, S.K., Salib, P.F., and Sy, O.**, "Exploitation Opportunities for the Oil Shales of Eastern Canada", presented at the CIM meeting, St. Johns, Newfoundland, November 1984.

7. **Salib, P., Barua, S. and Abbott, D.**, "Demonstration of an Integrated Oil Shales Retort System", Final Report, RPC report AS/85/51, 1984.

8. **Salib, P. and Boorman, R.S.**, "Research and Development Strategy for Co-Utilization of New Brunswick Coal and Oil Shale", RPC report MDP/83/7, 1983.

9. **Salib, P., Gilders, R. and Boorman, R.S.**, "Demonstration: Retorting of New Brunswick Oil Shales in a Pilot Scale Reator", RPC report MDP/83/16, 1983.

10. **Boorman, R.S., Salib, P., Gilders, R., and Gemmell, D.E.**, "Fluidized Bed Co-Combustion of Coal and Oil Shales a Route to SO_2 Emission Control", Paper presented at the 3rd Meeting of CIM District 1, Fredericton, Sept. 1982.

11. **Salib, P., and Swithenbank, J.**, "Pseduo-Random Stimulus Response of a Rotating Fluidized Bed Combustor", Paper presented at the AICHE 74th Annual Meeting, New Orleans, Nov. 1981.

12. **Turan, A., Swithenbank, J., Billings, S.A., and Salib, P.**, "Pseudo-Random Stimulus Response of Combustion Systems", Journal of Institute of Energy Dec, 1980, p. 181-190.

13. **Salib, P., Swithenbank, J., and Demircan, N.**, "The Rotating Fluidized Bed as a High Intensity Combustor", 3rd Int. Conf. Mech. Power Engng., Monofia University, Egypt, Sept. 1980.

CURRICULUM VITAE

RESEARCH AND PRODUCTIVITY COUNCIL

ROSS D. GILDERS

Education

1975 B.Sc. (Hon) University of Ottawa, Geology

Specialties

Process Metallurgy, Applied Mineralogy, Automated Image-Analysis

Experience

The New Brunswick Research and Productivity Council, Fredericton, NB. Mineral Development and Processing Laboratory and Semi Pilot Plant Process Research and Development in Fluid Bed Technology, Directed Towards Sulphation Roasting of Complex Base Metal Ores.

Senior Fluid Bed Roaster Operator and Maintenance Engineer, Member of Pyrometallurgical System Design Group.

Design and construction of a 6" fluidized bed test facility for fuming of arsenic trioxide. System includes high temperature baghouse, condenser and cold baghouse.

Fuming of crude arsenic baghouse dust for Giant Yellowknife Mines Ltd. for recovery of gold and high grade arsenic trioxide.

Investigation of sulphur capture during circulating fluidized bed combustion of Eastern Canadian Coal.

Chatham Sulphation Roast-Leach Pilot Plant; Process Design; Process/ Mechanical Detail Design; procurement Coordination. Planning and Scheduling; Equipment Design and Operating Specifications; Equipment Cost Projection. Plant Superintendent- Pyrometallurgy.

Technical evaluation, scrap lead-acid battery recycling process.

Economic evaluation of a proposed scrap lead-acid battery recycling business.

Plant visit and process evaluation, scrap lead acid battery plant.

Preliminary evaluation of a scrap automobile catalytic converter recycling business for platinum group metals recovery.

High Temperature/Pressure Pyrolysis Design Engineer.

Arsenic Removal from Copper Concentrates by Fluid Bed Roasting.

Anaconda Copper Company, RPC Sulphation Roast Process Trials and Application, Caribou Ore and Bulk Concentrate.

Examination of Off Gas Cleaning and Cooling System for the RPC Sulphation Roast-Leach Electrowinning Process.

RPC Sulphation Roast Process, Development and Application to new Brunswick Complex Base Metal Ores.

Bench Scale Sulphation Roasting Demonstration Trials on Brunswick - Type Mill Products.

Resource Utilization Linkage Program Zinc/Electrolytic Manganese Dioxide - CIDA/Indonesia Project.

The Technical Evaluation of the Application of the RPC Metals Recovery Process to Indonesian Zinc, Bulk Zinc-Lead, and Low Grade Manganese Concentrates.

Demonstration: Retorting of New Brunswick Oil Shales in a Pilot Scale Retort.

Fluidized Bed Combustion of High Sulphur Coal and Oil Shale.

Recovery of Gold and Silver via Segregation Roasting.

Selective Ferric Sulphate Oxidation/Brine Leach Process for the Recovery of Lead, Silver, and Zinc from Lead Cleaner Concentrate.

Development of Chloride Leach Process for Recovery of Lead and Associated Silver in Lead Concentrates.

Volume Reduction of Radioactive Waste by Steam Pyrolysis.

Calcination and Slaking Tests of a Limestone Sample.

Thermal Dissociation of Limestone to Lime.

Influence of the Mineralogy of Coal and in Particular of Sulphur Compounds on the Possibility of Sulphur and Ash Reduction.

Microscopic Investigation, Microsizing and Image Analyses of Coal Slurry Product.

Numerous Confidential Reports on Mineralogy of Coal and Base Metal Ores and Concentrator Mill Products.

Silver Carriers in the Brunswick Mining and Smelting Ore.

Distribution of Silver Carriers in Copper and Lead Circuit Tails.

Mineralogy Study of Aznalcollar Mill Samples.

Silver Carriers in Brunswick Concentrates and Possible Enhanced Recovery.

A preliminary Examination of Coke Fines at Brunswick Mining and Smelting.

The Mineralogy, Magnetic and Solution Behaviour of Select Mount Pleasant Tungsten Concentrates.

Silver Carriers in Concentrates and tailings from Brunswick Mining and Smelting Corporation Ltd.

Improved Silver Recovery at Brunswick Mining and Smelting.

Consultant to Minuvar Ltd. in Mineral Exploration and Development.

Immobilization of Complexed Metals Prior to Storage of Spent Ion Exchange Resins.

Sampling Campaign and Metal Distribution Study for Brunswick Mining and Smelting Ltd.

Comprehensive Mineralogical Survey of the Brunswick Mining and Smelting Mill and Concentrator.

Acid Generating Capacity of Decline Rock for Brunswick Mining and Smelting Ltd.

Mineralogy and Given Size Distribution of Ilmenite/Magnetite Ore.

Acid Generation by Waste Rock.

Mineralogical Investigation of 18 Concentrator Samples for East Kemptville tin Mines Ltd.

Publications

1. **Chalkley, M.E., Gilders, R.D., Jibiki, K., Synnott, J., Delaney, I.J.I., Grant, H.R., Boorman, R.S.,** The RPC Sulphation Roast Leach Process Pilot Plant Project, Technical Evaluation and Commercial Plant Scale Up. Presented to the CIM, 18th Hydrometallurgical Meeting, May 9-11, 1988.

2. **Synnott, J., Salter, R.S., Gilders, R.D., Chalkley, M.E.,** Iron Control In the RPC Sulphation-Roast-Leach Process, Proceedings International Symposium on Iron Control in Hydrometallurgy, Toronto, Ontario, Canada, Oct. 19-22, 1986.

3. **Salter, R.S., Synnott, J., Gilders, R.D., Doucet, G., and Boorman R.S.,** Design, Construction and Commissioning of the Demonstration Plant for the RPC Sulphation Roast Process, International Symposium on Complex Sulphides, November 10-13, 1985.

4. **Chryssoulis, S., Gilders, R.D., Surges, L.J., Salter, R.S. and Boorman, R.S.,** Silver Mineralogy at Brunswick Mining and Smelting and its Implications for Enhanced Recovery. Presented to the 23rd Annual Conference of Metallurgists, August 19-22, 1984.

5. **Sedman, K.G., Thornley, J.C., Gilders, R.D.,** The Validity of Fuel Recycling During Erosivity Testing Using Coal Water Liquid Fuels, ASTMA Symposium, Denver, Colorado, June 26-27, 1984.

6. **Stirling, A.R., Gilders, R.D.,** The Petrography of Pyrite in Some Maritime Coals. Atlantic Geoscience Symposium, Amherst, Nova Scotia, Survey 20-21, 1983.

7. **Salter, R.S., Boorman, R.S., Gilders, R.D.,** Chloride Leach Process for the Recovery of Lead and Silver Minerals and Process Residues. Canadian and United States Patents, 1982.

8. **Boorman, R.S., Salib, P., Gilders, R.D.,** Fluidized Bed Co-Combustion of Coal and Oil Shales, A Route to SO_2 Emission Control. Presented to the CIM 3rd District No. 1 Meeting, Sept. 21-25, 1982.

9. **Boorman, R.S., Petruk, W., Gilders, R.D.,** Silver Carriers in Concentrates and tailings from Brunswick Mining and Smelting Corporation Ltd. Presented to the CIM 3rd District No. 1 Meeting, Sept. 21-25, 1982.

10. **Gilders, R.D., Smith, P., Koster, E.H.,** Hydrologic and Hydrogeologic Effects of Urbanization. West Branch of Bilbery Creek and East Branch of Green's Creek, Orleans Ontario, 1976.

APPENDIX B

RAW DATA

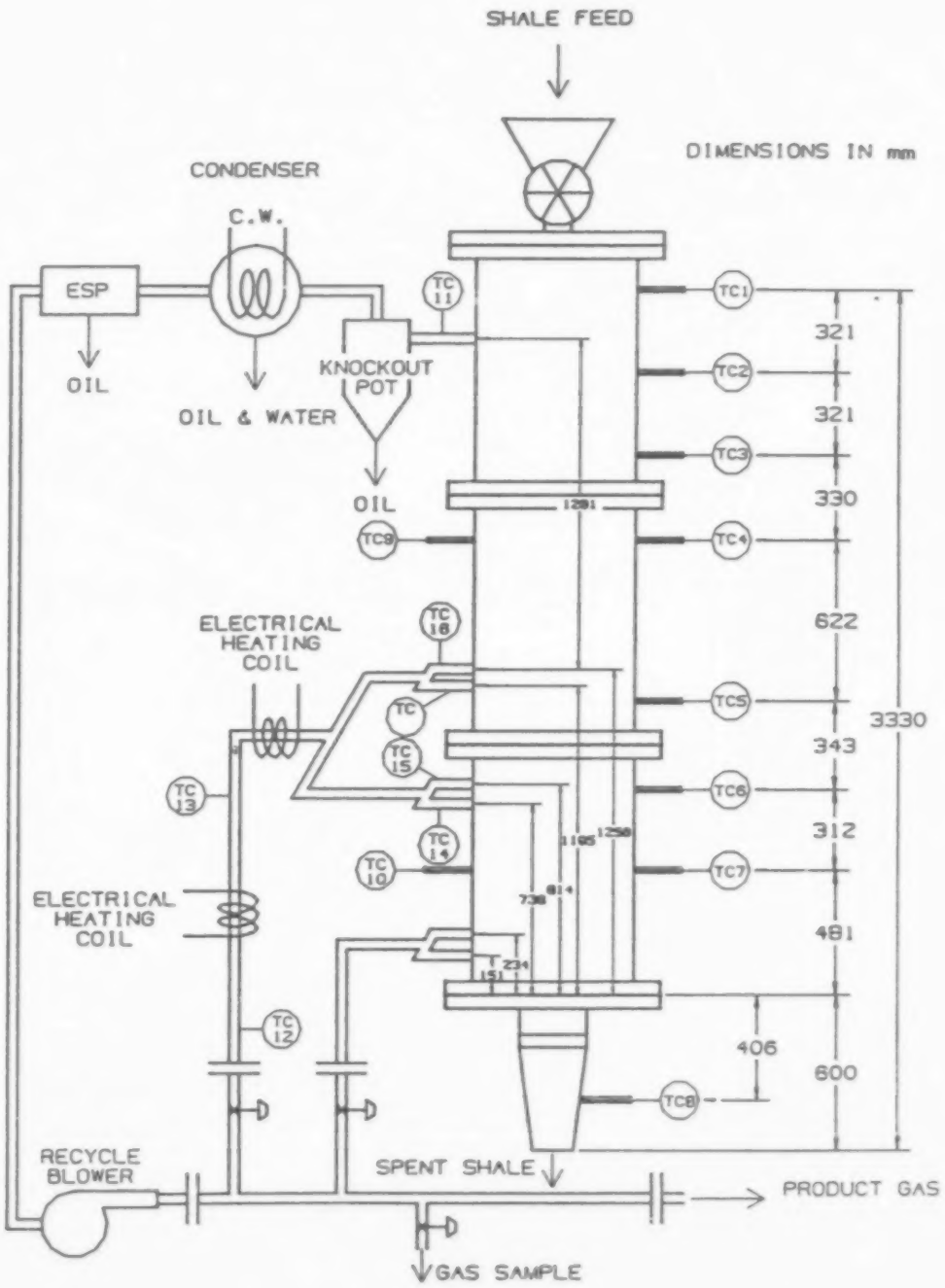
RAW DATA

The raw data recorded on an hourly basis for the two experimental runs are given in this Appendix. The data sheets are self-explanitory, however, the following clarifications may be useful.

- Feedrate = shale feedrate
- Discharge wt
  Discharge rate = spent shale discharge rate
  Discharge rate calc
- Temperatures T1 to T16 are an hourly average of temperatures recorded from thermocouples No. 1 to 16 as shown in Figure 1.
- At the end of each test run some statistical manipulation of the data are given. Those shown are the average, standard deviation, No. of points and minimum and maximum. Th statistical analysis were performed only on the last 18 hours of each test. The mean values were then used to calculate all the mass and energy balances given in the body of the report. The last eighteen hours were selected to be absolutely sure of steady state operation.

FIGURE 1

SCHEMATIC OF RPC FIXED BED RETORT SHOWING INSTRUMENTTION FOR TEMPERATURE AND FLOW MEASUREMENTS



RETORTING STUDY

Shale Type: Dolomite
Starting Date: 02/03/89
Starting Time: 11:00 a.m.
Finish Date: 03/03/89
Finish Time: 9:00 a.m.

DATE	D/M	02/3	02/3	02/3	02/3	02/3	02/3	02/3	02/3	02/3	02/3	02/3
TIME	H	11:00	12:00	13:00	14:00	15:00	16:00	17:00	18:00	19:00	20:00	21:00
FEED RATE	KG/H	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
DISCH WT	KG	68.20	81.80	101.60	114.80	131.80	143.40	159.80	30.80	45.80	60.60	76.60
DISCH RATE	KG/H	11.80	13.60	19.80	13.20	17.00	11.60	16.40				16.60
DISCH RATE CALC	KG/H		13.60	19.80	13.20	17.00	11.60	16.40		15.00		16.00
BED HEIGHT	IN	48.00	48.00	48.00	39.00	37.00	35.50	33.50	34.00	31.50	27.00	28.00
TOT RECY GAS	"H2O	23.00	22.50	23.00	23.50	24.50	23.00	23.00	21.50	22.00	23.00	23.50
	PSIG	6.40	4.30	4.30	4.70	5.00	4.60	4.60	3.50	3.20	3.80	3.80
MID RECY GAS	"H2O	9.00	8.00	8.00	9.00	10.00	8.50	8.50	6.50	7.00	9.00	9.00
	PSIG	1.90	2.00	1.90	2.10	2.20	2.20	2.20	1.10	1.10	2.00	2.10
BOT RECY GAS	% FLOW	5.30	6.00	5.10	5.20	6.00	6.00	4.90	5.90	5.20	5.00	5.00
	PSIG	0.30	0.50	0.50	0.50	0.60	0.60	0.60	0.40	0.50	1.00	1.00
VENT GAS	% FLOW											
	PSIG											
PURGE AIR	% FLOW	11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.50	11.50
	PSIG	11.70	11.60	12.00	11.80	11.80	11.90	12.00	12.00	11.70		11.60
VACUUM	"H2O	8.00	0.00	3.00	3.00	4.00	1.00	3.00	8.00	7.00	10.00	10.00
	KG											
KNOCKOUT OIL	ML	300	230	215	310	550	250	175	80	170	150.00	200.00
TOTAL COND	ML	1210	1250	1500	1230	1420	1380	1270	1080	1000	-	
COND H2O	ML	700	750	810	880	820	880	720	770	740	790	850
COND OIL	ML	510	500	690	350	600	500	550	310	260	330	250
ESP OIL	ML	1000	870	1220	920	1220	1240	960	790	670	970	950
OTHER OIL	ML	110	90	55	70	60	90	60	70	120	25	20
TOTAL OIL	ML	1920	1620	2180	1650	2430	2080	1745	1250	1220	1580	1520
TOTAL OIL CALC	ML	1920	1690	2180	1650	2430	2080	1745	1250	1220	1475	1420
TEMPERATURES		11:00	12:00	13:00	14:00	15:00	16:00	17:00	18:00	19:00	20:00	21:00
TOP(T)	T1	207	168	208	144	184	159	187		180	210	190
TOP(M)	T2	329	245	336	219	297	245	284		239	336	338
TOP(B)	T3	330	241	357	238	405	426	420		427	428	440
MID(T)	T4	508	500	447	482	506	520	526		516	535	542
MID(B)	T5	518	511	544	516	522	518	532		523	532	540
BOT(T)	T6	566	556	571	563	560	556	605		605	593	597
BOT(M)	T7	525	504	541	506	492	492	512		523	530	543
BOT(CONE)	T8	79	81	78	120	74	73	73		70	76	78
MID(T+1")	T9	440	442	413	433	447	451	457		453	461	464
BOT(M+1")	T10	433	430	437	435	430	433	443		440	446	449
GAS EXIT	T11	242	226	280	188	258	168	244		186	255	230
RECY GAS	T12	59	56	56	57	56	57	55		57	55	56
PREH BLAST	T13	366	369	373	369	385	379	397		386	382	379
LL RECY GAS	T14	602	609	608	609	603	598	648		636	607	605
RL RECY GAS	T15	560	563	560	561	557	550	579		583	564	562
RU RECY GAS	T16	598	603	601	603	599	594	641		632	603	603
RECY GAS AVG		587	592	590	591	586	581	623		617	591	590
GAS COMP												
CO2	%	9.00	9.90	10.00	10.20	10.00	8.00	10.00	11.80	11.70	12.00	
O2	%	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.00	0.00	
CO	%	0.30	0.20	0.30	0.30	0.20	0.20	0.20	0.20	0.20	0.20	
SO2	PPM											
NOx	PPM											
H2	%											
CH4	%											
SAMPLES												
OIL												
WATER												
SPENT SHALE												
GAS												

DATE	M/D	02/3	02/3	02/3	03/3	03/3	03/3	03/3	03/3	03/3	03/3	03/3
TIME	H	22:00	23:00	24:00	1:00	2:00	3:00	4:00	5:00	6:00	7:00	8:00
FEED RATE	KG/H	20.00	20.00	10.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
DISCH WT	KG	90.20	106.80	122.80	139.20	165.60	187.60	209.00	9.80	21.60	33.80	50.60
DISCH RATE	KG/H	13.60	16.60	16.00	16.40	26.40	12.00	21.40	9.80	11.80	12.30	16.80
DISCH RATE CALC	KG/H	13.60	16.60	16.00	16.40	26.40	22.00	21.40		11.80	12.20	
BED HEIGHT	IN	26.00	27.00	27.00	31.00	36.00	36.00	44.00	43.00	42.00	43.00	41.00
TOT RECY GAS	"H2O	23.00	22.00	22.00	21.00	22.00	22.00	22.00	22.50	23.00	22.50	22.00
	PSIG	3.80	5.50	5.40	5.00	6.20	6.20	6.20	6.50	7.20	7.20	7.30
MID RECY GAS	"H2O	9.00	8.00	8.00	7.50	9.00	9.00	9.00	9.00	8.50	8.50	8.00
	PSIG	2.10	2.00	2.00	1.80	2.00	2.00	2.00	2.10	2.10	2.20	2.20
BOT RECY GAS	% FLO	5.00	4.00	4.00	3.50	4.00	4.00	3.80	5.00	4.30	4.30	4.20
	PSIG	1.00	0.80	0.80	0.80	0.80	1.00	0.80	1.00	1.20	1.20	1.20
VENT GAS	% FLOW											
	PSIG											
PURGE AIR	% FLO	11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.00	
	PSIG	11.60	12.00	12.00	11.80	11.50	11.70	11.70	11.60	11.60	11.60	
VACUUM	"H2O	10.00	8.00	10.00	9.00	9.00	8.00	7.00	5.00	+2	+1	0.00
	KG											
KNOCKOUT OIL	ML	100.00	190.00	130.00	180.00	20.00	110.00	150.00	150	70	150.00	265
TOTAL COND	ML											
COND H2O	ML	850	890	700	730	930		700	790	690	1000	1190
COND OIL	ML	250	280	290	250	340	320	320	300	320	50	250
ESP OIL	ML	850	930	900	980	1020	1050	1060	1080	980	1030	1290
OTHER OIL	ML	10						150			220	200
TOTAL OIL	ML	1210	1400	1320	1410	1380	1480	1680	1530	1370	1400	2005
TOTAL OIL CALC	ML	1210	1400	1320	1410	1380	1480	1680	1530	1370	1450	2005
TEMPERATURES		22:00	23:00	24:00	1:00	2:00	3:00	4:00	5:00	6:00	7:00	8:00
TOP(T)	T1	192	196	192	209	198	205	198	204	186	211	193
TOP(M)	T2	358	401	409	333	302	307	300	293	293	321	272
TOP(B)	T3	442	435	440	437	393	357	305	294	256	290	190
MID(T)	T4	539	551	539	537	500	507	507	508	515	521	515
MID(B)	T5	536	544	540	544	539	533	539	524	530	529	527
BOT(T)	T6	596	588	586	611	592	584	598	560	561	571	565
BOT(M)	T7	555	547	537	550	565	557	566	531	523	912	519
BOT(CONE)	T8	87	91	96	108	174	142	149	134	102	92	
MID(T+1")	T9	464	471	465	463	450	448	447	464	467	469	466
BOT(M+1")	T10	449	453	450	454	467	463	460	461	457	450	455
GAS EXIT	T11	244	244	231	268	246	249	221	241	231	235	221
RECY GAS	T12	56	56	59	58	61	63	63	63	63	63	63
PREH BLAST	T13	382	394	393	408	414	386	392	420	395	396	377
LL RECY GAS	T14	606	605	605	640	637	605	609	594	597	602	599
RL RECY GAS	T15	560	562	564	577	640	572	565	550	551	557	556
RU RECY GAS	T16	600	601	604	626	635	603	607	588	595	599	596
RECY GAS AVG		589	589	591	614	637	593	594	577	581	586	584
GAS COMP												
CO2	%		9.80	8.80	9.00	12.40	10.00	11.80	9.80	10.00	10.00	14.2
O2	%		0.00	0.10	0.00	0.00	0.00	0.10	0.00	0.00	0.00	0.00
CO	%		0.50	0.30	0.30	0.30	0.30	0.30	0.40	0.30	0.30	0.30
SO2	PPM											
NOx	PPM											
H2	%											
CH4	%											
SAMPLES												
OIL												
WATER												
SPENT SHALE												
GAS												

DATE	D/M	03/3	03/3	AVG	STD	CNT	MIN	MAX
TIME	H:M	9:00	0:00					
FEED RATE	KG/H	20.00	20.00	19.4	2.3	18	10.0	20.0
DISCH WT	KG	64.80	82.80	92.1	58.2	18	9.8	209.0
DISCH RATE	KG/H	14.20	18.00	16.9	4.0	15	9.8	26.4
DISCH RATE CALC	KG/H	14.20	18.00	21.1	10.0	14	11.8	44.4
BED HEIGHT	IN	40.00	39.00	35.3	6.1	18	27.0	44.0
TOT RECY GAS	"H2O	22.50	22.50	22.3	0.6	18	21.0	23.5
	PSIG	9.90	10.90	5.9	2.1	18	3.2	10.9
MID RECY GAS	"H2O	9.50	8.00	8.4	0.8	18	6.5	9.5
	PSIG	2.20	2.20	2.0	0.3	18	1.1	2.2
BOT RECY GAS	% FLO	5.00	5.90	4.6	0.7	18	3.5	5.9
	PSIG	1.20	1.40	0.9	0.3	18	0.4	1.4
VENT GAS	% FLOW							
	PSIG							
PURGE AIR	% FLO	11.00	11.00	11.1	0.2	17	11.0	11.5
	PSIG	12.00	14.00	11.2	2.9	17	0.0	14.0
VACUUM	"H2O	3.00	1.00	6.0	3.7	18	0.0	10.0
	KG							
KNOCKOUT OIL	ML	75	105	137.2	56.1	18	20	265
TOTAL COND	ML		1030	1095.0	105.0	4	1000	1270
COND H2O	ML	830	730	817.6	126.2	17	690	1190
COND OIL	ML	340	300	295.0	88.8	18	50	550
ESP OIL	ML	1150	970	979.4	131.2	18	670	1290
OTHER OIL	ML	75	30	89.1	70.2	11	10	220
TOTAL OIL	ML	1640	1405	1474.7	195.7	18	1210	2005
TOTAL OIL CALC	ML	1640	1405	1466.1	193.4	18	1210	2005
TEMPERATURES		9:00	10:00	AVG	STD	CNT	MIN	MAX
TOP(T)	T1	191	213	197.5	9.5	17	180	213
TOP(M)	T2	269	329	316.7	43.2	17	239	409
TOP(B)	T3	333	417	370.8	76.3	17	190	442
MID(T)	T4	521	526	523.8	14.3	17	500	551
MID(B)	T5	529	533	533.8	6.3	17	523	544
BOT(T)	T6	557	572	584.8	16.8	17	557	611
BOT(M)	T7	517	516	535.5	18.0	17	512	566
BOT(CONE)	T8	78	78	95.8	36.0	17	0	174
MID(T+1")	T9	465	465	461.1	7.2	17	447	471
BOT(M+1")	T10	457	460	453.8	7.1	17	440	467
GAS EXIT	T11	224	245	236.2	17.4	17	186	268
RECY GAS	T12	67	67	60.3	3.9	17	55	67
PREH BLAST	T13	382	400	393.1	11.9	17	377	420
LL RECY GAS	T14	593	605	611.4	16.7	17	593	648
RL RECY GAS	T15	553	561	568.0	20.2	17	550	640
RU RECY GAS	T16	593	602	607.5	15.3	17	588	641
RECY GAS AVG		580	589	595.6	16.3	17	577.3	637.3
GAS COMP								
CO2	%	9.20	12.20	9.9	2.8	16	0.0	12.4
O2	%	0.00	2.30	0.2	0.6	16	0.0	2.3
CO	%	0.20	0.30	0.3	0.1	16	0.2	0.5
SO2	PPM							
NOx	PPM							
H2	%							
CH4	%							
SAMPLES								
OIL								
WATER								
SPENT SHALE								
GAS								

RETORTING STUDY

Shale Type: Albert

Starting Date: 03/03/89

Starting Time: 11:00 a.m.

Finish Date: 04/03/89

Finish Time: 10:00 a.m.

DATE	D/M	03/3	03/3	03:3	03/3	03/3	03/3	03/3	03/3	03/3	03/3	03/3
TIME	H	11:00	12:00	13:00	14:00	15:00	16:00	17:00	18:00	19:00	20:00	21:00
FEED RATE	KG/H	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
DISCH WT	KG	111.20	130.80	153.20	31.40	50.60	70.00	85.40	102.60	14.20	134.80	149.00
DISCH RATE	KG/H	28.40	19.00	22.40		19.20	19.40	15.40	17.20	18.60	13.60	14.20
DISCH RATE CALC	KG/H	28.40	19.60	22.40		19.20	19.40	15.40	17.20			14.20
BED HEIGHT	IN	41.00	44.00	43.50	41.00	41.50	40.50	41.00	40.00	40.00	43.00	38.00
TOT RECY GAS	"H2O	22.50	22.50	23.00	21.00	22.00	21.50	22.50	23.00	22.50	22.00	22.00
	PSIG	11.20	10.00	9.30	11.40	10.80	10.20	8.60	8.90	8.70	9.30	9.40
MID RECY GAS	"H2O	8.00	10.00	10.00	9.00	9.00	8.50	9.00	9.50	9.00	10.00	10.50
	PSIG	2.20	2.30	2.60	2.20	2.20	2.00	2.20	2.40	2.20	2.30	2.40
BOT RECY GAS	% FLOW	4.90	5.20	5.00	4.30	5.00	4.30	4.10	4.10	4.20	5.10	5.10
	PSIG	1.20	1.20	1.20	1.20	1.00	1.10	1.10	1.10	1.10	1.00	1.00
VENT GAS	% FLOW											
	PSIG											
PURGE AIR	% FLOW	11.00	11.00	11.00	11.00	11.00	11.00	11.00	12.00	11.00	12.00	12.00
	PSIG	13.70	13.60	14.20	14.30	13.90	14.00	13.60	14.00	13.80	14.00	14.10
VACUUM	"H2O	1.00	0.00	2.00	4.00	7.00	6.50	6.00	8.00	7.50	7.00	8.00
	KG											
KNOCKOUT OIL	ML	90.00	300.00	100.00	340.00	350.00	400.00	340	550	370	350	375
TOTAL COND	ML											
COND H2O	ML	860	650	520	660	970	500	580	1030	1100	570	675
COND OIL	ML	260	870	1010	860	630	960	940	500	450	830	860
ESP OIL	ML	1470	2470	1930	1700	1830	1880	1800	2180	2120	2030	1960
OTHER OIL	ML	100	50	25	60	70	30	20	30	60	20	
TOTAL OIL	ML	1920	3690	3065	2960	2880	3270	3100	2710	3000	3230	3195
TOTAL OIL CALC	ML	1920	3690	3065	2960	2880	3270	3100	3260	3000	3230	3195
TEMPERATURES		11:00	12:00	13:00	14:00	15:00	16:00	17:00	18:00	19:00	20:00	21:00
TOP(T)	T1	188	228	190	209	185	186	175	176	211	177	167
TOP(M)	T2	265	332	301	321	301	304	267	250	319	298	250
TOP(B)	T3	262	337	277	330	301	273	295	344	428	434	461
MID(T)	T4	502	520	511	539	578	561	573	566	575	590	570
MID(B)	T5	540	519	522	539	557	559	552	566	551	566	555
BOT(T)	T6	580	561	557	583	601	573	599	602	586	600	572
BOT(M)	T7	564	532	520	509	563	544	544	546	541	548	542
BOT(CONE)	T8	120	129	107	101	122	107	103	115	109	114	104
MID(T+1")	T9	436	452	460	468	488	494	497	492	494	502	497
BOT(M+1")	T10	474	454	452	441	455	449	453	456	454	451	455
GAS EXIT	T11	237	257	217	208	204	209	195	169	211	207	183
RECY GAS	T12	69	65	64	66	65	66	65	64	63	66	66
PREH BLAST	T13	395	381	370	371	379	378	367	369	369	359	355
LL RECY GAS	T14	606	589	581	599	602	589	596	592	589	600	578
RL RECY GAS	T15	560	554	546	558	562	551	557	554	551	560	543
RU RECY GAS	T16	605	589	577	598	600	592	595	591	589	599	576
RECY GAS AVG		590	577	568	585	588	577	583	579	576	586	566
GAS COMP												
CO2	%	12.00	7.00			10.00	11.20	14.20	13.00	12.20	13.00	10.60
O2	%	2.30	2.40			0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	%	0.40	0.40			1.00	0.60	1.20	0.70	0.70	0.70	0.60
SO2	PPM											
NOx	PPM											
H2	%											
CH4	%											
SAMPLES												
OIL												
WATER												
SPENT SHALE												
GAS												

DATE	D/M	03/3	03/3	03/3	04/3	04/3	04/3	04/3	04/3	04/3	04/3	04/3
TIME	H	22:00	23:00	24:00	1:00	2:00	3:00	4:00	5:00	6:00	7:00	8:00
FEED RATE	KG/H	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
DISCH WT	KG	164.20	181.00	6.80	18.20	40.80	56.60	73.00	88.40	96.40	114.40	129.80
DISCH RATE	KG/H	15.20	16.80		11.40	22.60	15.80	16.40	15.40	8.00	18.00	15.40
DISCH RATE CALC	KG/H	15.20	16.80		11.40	22.60	15.80	16.40	15.40	8.00	18.00	15.40
BED HEIGHT	IN	38.00	39.00	39.00	40.00	40.00	39.00	38.00	38.00	37.00	38.00	37.00
TOT RECY GAS	"H2O	22.00	22.00	23.00	23.00	23.00	23.50	23.50	24.00	24.00	24.00	23.50
	PSIG	9.30	9.10	9.80	8.10	8.80	8.00	7.80	7.50	7.50	7.00	7.00
MID RECY GAS	"H2O	10.00	10.00	10.00	9.00	10.00	8.50	9.00	9.50	9.00	9.00	9.00
	PSIG	2.50	2.50	2.90	2.20	2.40	2.30	2.10	2.20	2.20	2.30	2.30
BOT RECY GAS	% FLO	5.00	5.00	5.00	5.20	5.40	5.00	5.00	5.00	4.50	5.00	5.80
	PSIG	1.00	1.00	1.20	1.00	1.30	1.20	1.00	1.00	1.00	1.00	1.10
VENT GAS	% FLOW					42.00	36.00	34.00	33.00	33.00	32.00	33.00
	PSIG					0.80	0.50	0.50	0.50	0.50	0.50	0.70
PURGE AIR	% FLO	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00
	PSIG	14.00	14.10	14.30	14.10	14.00	14.00	13.80	14.00	13.60	13.60	13.80
VACUUM	"H2O	6.50	6.00	3.00	6.00	0.00	2.00	7.00	5.00	7.00	5.00	5.00
	KG											
KNOCKOUT OIL	ML	360	235	330	275	120	20	690	190	190	250	230.00
TOTAL COND	ML											
COND H2O	ML	520	500	640	770	810	720	800	620	690	550	620
COND OIL	ML	850	920	980	800	640	680	800	970	920	920	940
ESP OIL	ML	2260	2310	2330	2450	2090	1920	2340	2240	2270	2160	2270
OTHER OIL	ML		40			30					30	
TOTAL OIL	ML	3470	3505	3640	3525	3050	2620	3830	3050	3880	3360	3440
TOTAL OIL CALC	ML	3470	3505	3640	3525	2880	2620	3830	3400	3380	3360	3440
TEMPERATURES		22:00	23:00	24:00	1:00	2:00	3:00	4:00	5:00	6:00	7:00	8:00
TOP(T)	T1	162	170	183	191	170	162	178	172	180	177	208
TOP(M)	T2	251	304	310	317	250	246	324	308	318	320	335
TOP(B)	T3	459	455	448	452	442	433	468	483	481	466	463
MID(T)	T4	580	583	571	555	574	605	596	605	589	590	584
MID(B)	T5	551	555	557	542	549	569	583	590	569	568	567
BOT(T)	T6	575	586	572	552	574	612	591	600	575	584	582
BOT(M)	T7	533	537	550	514	517	527	528	526	526	529	528
BOT(CONE)	T8	101	105	108	87	78	83	92	84	87	84	83
MID(T+1")	T9	495	497	495	485	485	492	500	498	501	503	501
BOT(M+1")	T10	449	447	450	445	438	447	446	446	448	450	455
GAS EXIT	T11	183	218	220	222	188	181	216	219	219	216	222
RECY GAS	T12	65	65	66	65	63	61	61	61	61	61	59
PREH BLAST	T13	363	369	361	378	375	384	382	374	373	380	377
LL RECY GAS	T14	585	593	580	574	596	611	614	604	594	597	605
RL RECY GAS	T15	547	553	546	536	555	567	578	563	553	557	561
RU RECY GAS	T16	584	591	578	575	595	613	610	603	593	600	603
RECY GAS AVG		572	579	568	562	582	597	601	590	580	585	590
GAS COMP												
CO2	%	10.60	11.20	10.00	8.40	12.60	12.60	12.90	14.00	12.00	12.80	13.00
O2	%	0.00	0.00	0.00	0.00	0.10	0.00	0.10	0.00	0.00	0.10	0.00
CO	%	0.70	0.70	1.50	0.70	2.00	1.00	0.60	0.50	0.70	0.80	0.70
SO2	PPM											
NOx	PPM											
H2	%											
CH4	%											
SAMPLES												
OIL												
WATER												
SPENT SHALE												
GAS												

DATE	D/M	04/3	04/3	AVG	STD	CNT	MIN	MAX
TIME	H	9:00	10:00					
FEED RATE	KG/H	20.00		20.0	0.0	17.0	20.0	20.0
DISCH WT	KG	149.80	17.20	90.1	53.9	18.0	6.8	181.0
DISCH RATE	KG/H	20.00		15.9	3.2	16.0	8.0	22.6
DISCH RATE CALC	KG/H	20.00		15.8	3.3	14.0	8.0	22.6
BED HEIGHT	IN	37.00		38.9	1.6	17.0	37.0	43.0
TOT RECY GAS	"H2O	21.50	23.00	22.9	0.8	18.0	21.5	24.0
	PSIG	10.30	8.10	8.5	0.9	18.0	7.0	10.3
MID RECY GAS	"H2O	9.50	10.00	9.5	0.5	18.0	8.5	10.5
	PSIG	2.60	2.60	2.4	0.2	18.0	2.1	2.9
BOT RECY GAS	% FLO	4.60	5.20	4.9	0.4	18.0	4.1	5.8
	PSIG	1.20	1.10	1.1	0.1	18.0	1.0	1.3
VENT GAS	% FLOW			34.7	3.2	7.0	32.0	42.0
	PSIG			0.6	0.1	7.0	0.5	0.8
PURGE AIR	% FLO	12.00	12.00	11.9	0.3	18.0	11.0	12.0
	PSIG	13.70	14.00	13.9	0.2	18.0	13.6	14.3
VACUUM	"H2O	1.00	5.00	5.3	2.3	18.0	0.0	8.0
	KG							
KNOCKOUT OIL	ML	220.00	220.00	295.3	147.8	18.0	20.0	690.0
TOTAL COND	ML							
COND H2O	ML	700	570	692.5	159.1	18	500	1100
COND OIL	ML	890	850	818.9	150.4	18	450	980
ESP OIL	ML	2270	2120	2173.3	161.6	18	1800	2450
OTHER OIL	ML			32.9	12.8	7	20	60
TOTAL OIL	ML	3380	3190	3287.5	331.2	18	2620	3880
TOTAL OIL CALC	ML	3380	3190	3300.3	275.1	18	2620	3830
TEMPERATURES		9:00	10:00	AVG	STD	CNT	MIN	MAX
TOP(T)	T1	204	184	180.4	14.2	18	162	211
TOP(M)	T2	333	261	292.3	32.3	18	246	335
TOP(B)	T3	479	247	429.9	64.2	18	247	483
MID(T)	T4	570	592	581.6	13.2	18	555	605
MID(B)	T5	561	561	561.8	11.7	18	542	590
BOT(T)	T6	569	595	584.8	14.6	18	552	612
BOT(M)	T7	517	538	532.8	10.7	18	514	550
BOT(CONE)	T8	76	124	96.5	14.0	18	76	124
MID(T+1")	T9	487	490	495.1	5.5	18	485	503
BOT(M+1")	T10	448	449	449.3	4.3	18	438	456
GAS EXIT	T11	221	194	204.7	17.1	18	169	222
RECY GAS	T12	65	64	63.4	2.1	18	59	66
PREH BLAST	T13	377	369	371.2	7.9	18	355	384
LL RECY GAS	T14	589	606	594.6	10.8	18	574	614
RL RECY GAS	T15	549	561	555.1	9.3	18	536	578
RU RECY GAS	T16	587	601	593.5	10.6	18	575	613
RECY GAS AVG		575	589	581.1	10.1	18	562	601
GAS COMP								
CO2	%	12.00	14.40	12.2	1.5	18	8.4	14.4
O2	%	0.00	0.10	0.0	0.0	18	0.0	0.1
CO	%	0.60	0.80	0.8	0.4	18	0.5	2.0
SO2	PPM							
NOx	PPM							
H2	%							
CH4	%							
SAMPLES								
OIL								
WATER								
SPENT SHALE								
GAS								

APPENDIX C

ASTM DISTILLATION

PROPOSED SIMULATED DISTILLATION METHOD

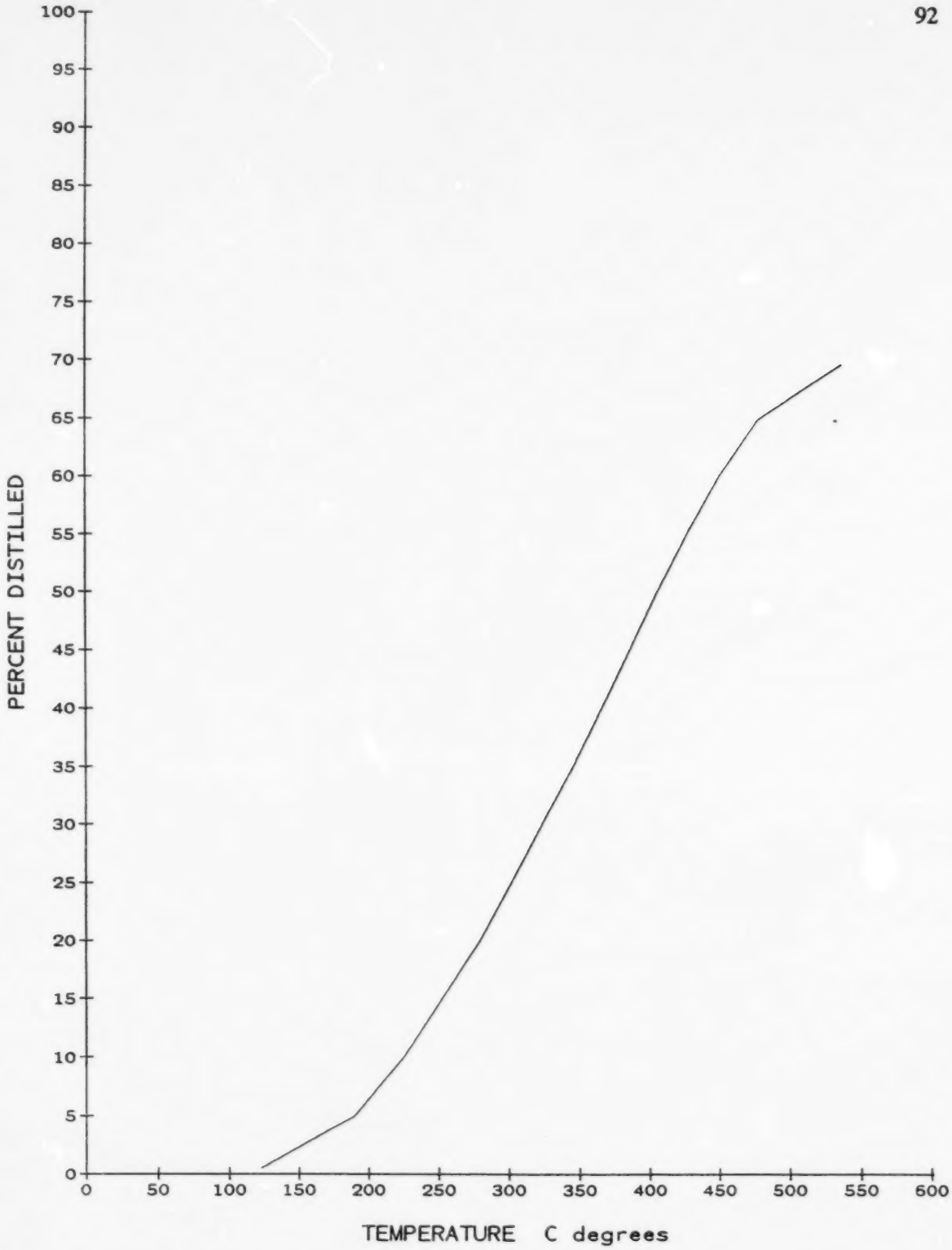
Sample Id.: Iota
Date:

Cumulative Percent Distilled = 69.8

% Off	Temp Deg C
0.5	123
5	190
10	225
15	252
20	279
25	301
30	323
35	345
40	365
45	385
50	405
55	426
60	449
65	477
69.8	536

COMPOSITE ALBERT SHALE OIL SAMPLE.

SIMULATED DISTILLATION ANALYSIS



Job Number: 5313 Sample Number: LOTA

PROPOSED SIMULATED DISTILLATION METHOD

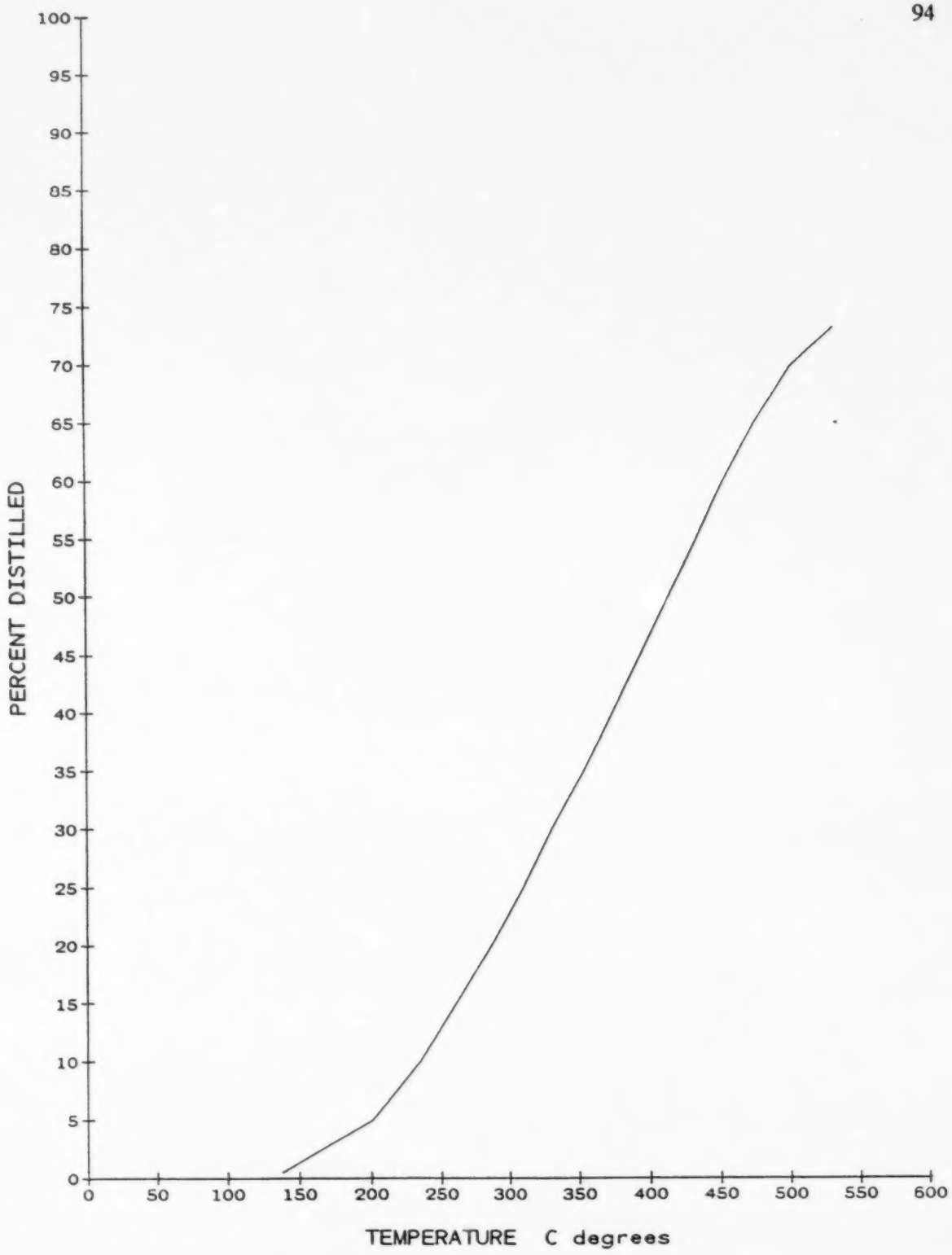
Sample Id.: lotb
Date:

Cumulative Percent Distilled = 73.4

% Off	Temp Deg C
0.5	138
5	201
10	235
15	261
20	287
25	310
30	331
35	354
40	374
45	394
50	414
55	434
60	453
65	475
70	502
73.4	533

COMPOSITE DOLOMITE ZONE SHALE OIL SAMPLE.

SIMULATED DISTILLATION ANALYSIS



Job Number: 5313 Sample Number: L0TB